March 1957 Pages 81 - 120

The Journal
of the
SOCIETY OF DYERS AND COLOURISTS

NEW COLOUR INDEX
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Volume 73 Number 3

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THE SOCIETY OF DYERS AND COLOURISTS
19 PICCADILLY BRADFORD 1 YORKSHIRE

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In view of the high costs of publication, it is essential that authors should be as concise as possible. When experimental procedure has already been published, a literature reference to the paper containing the details is sufficient, whilst well known experimental methods should be described very briefly.

Introductory paragraphs describing the aims of the investigation and the method of attack are desirable, and should be followed by the experimental results and their discussion. There should be a brief summary for insertion at the beginning of the paper. References to the literature should be numbered consecutively, using superscript numbers without brackets immediately following the text words or author's name to which they refer.

The list of references should be given at the end of the manuscript and the abbreviations used should be, as far as possible, those given in the "List of Periodicals Abstracted" included at the end of the index to the preceding year's *Journal*. Reference numbers in this list should be neither enclosed in brackets nor followed by full-stops. As far as possible throughout the manuscript the abbreviations listed in the Jan. 1950 issue (p. 54) should be used. Tables should be numbered consecutively in Roman numerals and Figures in Arabic numerals.

The number of figures and graphs should be kept as low as possible, and data should be presented in the form of either tables or graphs, not both. Drawings should be carefully prepared, preferably in indian ink, on plain white drawing paper or, preferably, Bristol board. In graphs, the frame and actual curves should be ruled and inked more heavily than any co-ordinate lines, and the latter should not be close together as in ordinary graph paper. Experimental points should always be given, and where several graphs appear in a single Figure clear means of differentiation must be adopted. All numbers and legends are set up in type by the printer, and authors should therefore indicate them lightly in pencil.

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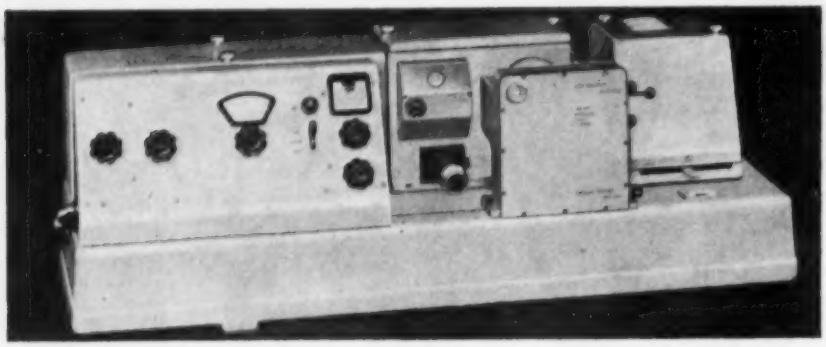

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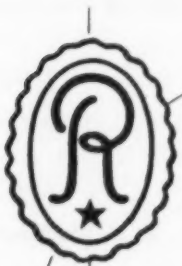

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Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal* —

LECTURES

Dyeing of Man-made Fibres in the Carpet Trade — *R. C. Cheetham*

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A Critical Appraisal of the International Daylight Fastness Test — *K. McLaren*

COMMUNICATIONS

The Dyeing of Cellulose Acetate with Disperse Dyes—VI — *C. L. Bird and Miss P. Harris*

Determination of the Migratory Properties of Direct Dyes — *J. Cegarra*

The Flameproofing of Nylon — *D. O. Douglas*

Light-fastness Assessments of Dyed Textiles and their Bearing upon the Mechanism of Fading — *C. H. Giles*

Alginate Hessian — *E. N. Woodward* et al.

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FORTHCOMING MEETINGS OF THE SOCIETY

Friday, 22nd March 1957

BRADFORD JUNIOR BRANCH. Annual Dance. Connaught Rooms, **Bradford.**

Thursday, 28th March 1957

WEST RIDING SECTION. Annual General Meeting. Victoria Hotel, Bridge Street, **Bradford.** 7.30 p.m.

Friday, 29th March 1957

The Society's Annual Dinner. The Grosvenor House, Park Lane, **London.**

Thursday, 4th April 1957

MIDLANDS SECTION. *The Soiling of Natural and Synthetic Fibres.* G. G. Taylor, Esq., B.Sc., A.Inst.P. (Joint with the Textile Institute.) Carpet Trades Canteen, **Kidderminster.** 7.30 p.m.

Friday, 5th April 1957

LONDON SECTION. *Some Aspects of Bleaching with Hydrogen Peroxide and Peracetic Acid.* L. Chesner, Esq., B.Sc., A.T.I. and G. C. Woodford, Esq. (Laporte Chemicals Ltd.). George Hotel, **Luton.** 7 p.m.

Tuesday, 9th April 1957

SCOTTISH SECTION. Annual General Meeting. 7 p.m. Followed by ordinary Meeting at 7.30 p.m. *Developments in Sizing as they Affect the Dyer and Finisher.* J. H. McGregor, Esq., Ph.D., F.R.I.C. and E. France, Esq., A.R.C.S., A.M.C.T. (Courtaulds Ltd.). St. Enoch Hotel, **Glasgow.**

Friday, 12th April 1957

LONDON SECTION. Annual General Meeting and Dinner. Aldwych Brasserie, Aldwych, **London W.C.2.** 6.30 p.m.

MIDLANDS SECTION. Annual Dinner. George Hotel, **Nottingham.** 7 p.m.

Wednesday, 17th April 1957

MIDLANDS SECTION. . . . . *and inwardly digest* (*a Dissertation on Technical Literature*). S. Burgess, Esq., F.S.D.C., A.M.C.T. (Preceded at 6.30 p.m. by the Annual General Meeting of the Section). King's Head Hotel, **Loughborough.** 7 p.m.

Saturday, 20th April 1957

BRADFORD JUNIOR BRANCH. Annual General Meeting. Technical College, **Bradford.** 10.15 a.m.

Friday, 3rd May 1957

MIDLANDS SECTION. The Mercer Lecture of the Society. Grand Hotel, **Leicester.** 7 p.m.

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THE JOURNAL
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Volume 73 Number 3 MARCH 1957 *Issued Monthly*

Proceedings of the Society

William Henry Perkin

JOHN BOULTON

Perkin Centenary Lecture of the Northern Ireland Section given at Thompson's Restaurant, Belfast, on 14th November 1956, Mr. R. J. G. Reid in the chair*

Amongst the mass of material that has appeared on the subject of William Perkin during the centenary year, a number of papers have been written and delivered on the effects of his discovery on the organic chemical industry generally, upon aspects of scientific and industrial development, such as dyes and dye chemistry, drugs and pharmacology, plastics and fibres, and the production of many new substances which have enriched our life. Much has been said about our debt to Perkin with regard to these material advances, by a number of authors and journalists, some more distinguished and knowledgeable than others. Browsing, myself, in some of the more densely worded acreages of this paper world, I have, as an invited lecturer on this subject, realised three things. In the first place — and this presents a difficulty to still another Centenary lecturer — almost everything that could be said about the material importance of Perkin's discovery, during the hundred years that have elapsed since he made it, has been said. Secondly, it is not often very easy for the modern chemist to appreciate the actual experiments that Perkin in fact did. The journalists, of course, have not understood them sufficiently well to give an account, and most of the distinguished scientific contributions have tended to take them for granted. Further, and perhaps most important, little has been said about the importance of the man himself as distinct from his discoveries.

I have been driven to reflect upon the question of what kind of man is a Perkin. How comes it that there are so few of them? What is it in their background and their make-up that drives them, or leads them, to do the outstanding things they do? Many readers of the *Journal* will have benefited already from the papers that have appeared throughout the centenary year. I will therefore content myself with a short account of Perkin's life, and see what we can learn by reflecting on the nature of the man in terms of the social, industrial, and intellectual scene as we experience it in our time.

William Henry Perkin, born in London on 12th March 1838, was one of George Fowler Perkin's seven sons. His father was at that time a builder and carpenter and the family had a practical background of Northern craftsman stock. There is also evidence that William's Yorkshire grandfather had an active interest in chemical experiments. The remains of his equipment were found in the cellar of his house near Ingleton. The family also had an artistic life, especially in the realms of music, a field in which William was particularly interested and capable. In the family group of nine instrumentalists, William Perkin played the double bass.

As a boy he was interested in carpentry and engineering and also had serious intentions at one time in his early youth of becoming a painter. His interest in painting stayed with him throughout his life. At his father's wish, these practical and artistic bents were to be combined in the study of architecture, but schoolboy experiments fired young William's imagination with an enthusiasm for chemistry, and this was, by the time he had reached the very early teens, decided upon as his career.

In his speech at the Dinner given to him in New York on 6th October 1906, to celebrate the coal-tar colour jubilee, Sir William Perkin said—

> My father being a builder, the first idea was that I should follow in his footsteps, and I used to watch the carpenters at work and also tried carpentering myself. Other things I noticed led me to take an interest in mechanics and engineering, and I used to pore over an old book called *The Artisan*, which referred to these subjects. I even tried to make an engine myself, and got so far as making the patterns for casting, but I was unable to go further for want of appliances. I had always been fond of drawing and sometimes copied plans for my father, whose ambition was that I might be an architect. This led me on to painting and made me think I should like to be an

* The account of Perkin's discovery of Mauve and its development, which have been frequently described elsewhere (*cf. J.S.D.C.*, **72**, 566 (1956)) has been drastically shortened.

> artist, and I worked away at oil painting for some time. All these subjects I pursued earnestly and not as amusements, and the information I obtained, though very elementary, was of much value to me afterwards. But when I was between 12 and 13 years of age, a young friend showed me some chemical experiments and the wonderful power of substances to crystallise in definite forms, and the latter, especially, struck me very much, with the result that I saw there was in chemistry something far beyond the other pursuits with which I had previously been occupied. The possibility also of making new discoveries impressed me very much. My choice was fixed, and I determined if possible to become a chemist, and I immediately commenced to accumulate bottles of chemicals and made experiments.

In Perkin's childhood chemistry was not taught as a regular school subject, except in very few schools indeed. One of these was the City of London School, where Perkin was a scholar, and even here it was taken in a spare-time class with two lessons a week given in the dinner interval, so that science should not interfere with the proper education of the young. William soon became the teacher's assistant, helping to assemble the apparatus in preparation for the lectures. One of Perkin's treasured possessions was a letter given by Michael Faraday in his own hand, providing Perkin with permission to attend the lectures on electricity at the Royal Institution in 1852.

At 15 years old Perkin entered the Royal College of Chemistry in Oxford Street, London, and studied under the first Professor there, A. W. Hofmann. By the time he was 17 he was helping in research projects. His first task was concerned with the nitration of anthracene, and although anthraquinone, not nitroanthracene, was the product of the reaction, the experience was to help him later, when he wished to manufacture synthetic alizarin. Such was his enthusiasm still, that work during the day was not enough and he set up a laboratory at home, so that his evenings and holidays could also be filled. It was here that, in an attempt to obtain quinine, Perkin oxidised allyltoluidine and obtained a dirty brown powder. Though he had produced no quinine, the young experimenter realised that the brown powdery mass which his efforts had produced represented some change in his starting materials and in it there might be something of value. He repeated the experiment with the simpler substance, aniline. This perseverance is particularly remarkable, since the temper of the age and his own inclination were to regard crystalline substances as the ultimate goal. However, it may be that the glimpse of colour also appealed to his artistic nature, making him press on to isolate the purple substance, whilst his practical bent induced him to try its power as a dye. Whatever his motive, he did persevere until he had produced enough of his Aniline Purple to dye some patterns and skeins of silk. These patterns were sent to Robert Pullar, the dyer of Perth, who was struck by the quality of the colour and its fastness and wrote encouragingly about it.

Thus we see that the discovery of Mauve was an accident: looking for one thing, Perkin found another.

It was at this point that the great decision was taken which may be said to have founded the synthetic-dye industry. In spite of lack of enthusiasm on the part of the calico printers and in the face of opposition from his teacher, Hofmann, who tried to influence him to remain a research student, Perkin had such faith in the new dye that he decided to go ahead with its manufacture, even though in many respects his own inclinations were in sympathy with the arguments of Hofmann. Difficulties arose at the outset from lack of backers to provide the capital, but soon his elder brother, Thomas Dix Perkin, had also given up architecture to join him, and their father gave his savings to finance the venture. The story of how the Greenford Works was built and brought into production is a fine example of tenacity and pioneering. Neither of the brothers had seen a chemical works, and even if they had, little of the equipment then in use was suitable for the processes involved in the production of the dye. They had perforce to become chemical engineers, and Thomas seems to have had quite a flair. Building was begun in June 1857, and by December Aniline Purple, or Tyrian Purple, was being used in the silk dyehouse of Thomas Keith & Sons at Bethnal Green.

During this time the second problem, that of raw material supplies, had also been solved. The oxidation process was carried out with potassium or sodium dichromate, which could be obtained fairly easily. Aniline, however, was still a rare laboratory chemical. A method for producing it by the reduction of nitrobenzene with iron borings and acetic acid was already known, but nitrobenzene was not available in bulk either. Benzene had been recognised as a constituent of coal tar some years earlier by Hofmann, but supplies were still difficult, and the only product obtainable was so crude that it required redistillation before it could be used. It was then thought that the nitration of benzene could be carried out only with fuming nitric acid, and this, too, was very scarce, and expensive. Perkin, therefore, began experiments using mixtures of nitric acid and sulphuric acid and also sodium nitrate and sulphuric acid. The second of these was put into large-scale production, the hazards of scale-effects, which cause the reaction to become explosive, being encountered and solved in the process.

Perkin's first dyeings had been made on silk, and the affinity of the dye for that fibre was particularly good; in fact, when the availability of greater quantities of the dye made bulk work possible, it was found that unlevel dyeings were produced. Turning his attention to this problem, Perkin discovered that the addition of soap to the dyebath gave much more even results, and this method became general in silk dyeing. The dyeing of silk, however, covered a very limited field, and it was essential for the success of the manufacturing project that the dye should be applicable to cotton, especially for printing. The dye itself had little affinity for cotton; the conservative English printers resisted attempts to introduce it, and it seemed as if failure was inevitable. Never content to let a challenge go unanswered, Perkin set out to find a method which would make calico printing with his dye possible.

At the same time Robert Pullar was also at work, and almost simultaneously these two men reached the conclusion that the way to success lay in first treating the cotton with a mordant such as tannin followed by sodium stannate or alum and then dyeing with the Aniline Purple. Further work on the printing process produced several improved methods, until finally one was perfected which could be used to give clear bright colours in multicolour prints.

The initial success had not only given the Perkins a good start but had also stimulated others to enter the field, and soon many new synthetic dyes were being produced. Perkin's own investigations led to the production of several more dyes at the Greenford Green works, including the triphenylmethane derivatives— Britannia Violet and Perkin's Green— and later synthetic alizarin. The alizarin stage marked the end of Perkin's direct contributions to the dyestuffs industry. By 1873 expansion of the Greenford Green works became necessary; instead of undertaking this the Perkin family sold the business, and from 1874, at the age of 36, William again gave his whole attention to research. Throughout the manufacturing period he had had his private laboratory, where he continued to follow his own lines of investigation. Indeed, it was during that period, in 1868, that he first made use of the reaction which every chemist today honours with his name. One application of this reaction led to coumarin, the first perfume to be produced synthetically.

During his later years William Perkin devoted himself largely to physicochemical problems, being particularly interested in magnetic rotatory power and its relationship to chemical constitution. He went on publishing papers, mainly in the *Journal of the Chemical Society*, until only a few months before his death.

The length of Perkin's working life was such that he was able to take part in the 1906 celebrations of the jubilee of his first famous discovery. In that year he was knighted, and in addition to the portrait* by A. S. Cope, which was given to him for the period of his life, and which is now in the National Gallery, medals and addresses of respect were presented to him by many English and foreign scientific societies. The address presented on behalf of the Society of Dyers and Colourists by the President, Thomas Wardle, is printed in full in the August 1906 issue of the *Journal*, and an account of the proceedings follows in the September issue. In 1907 Perkin himself became President of the Society, but he did not live to complete the full term of his office. After only a few days' illness he died on 14th July 1907.

It is on record that one member of Perkin's family said that it was not until the 1906 celebrations that they came to realise his high reputation: it would therefore appear that William was a man of unusual modesty inside the family circle.

I have recently been reading the views of distinguished literary men on the "two cultures" which, in this intensely scientific and technological age, are said to be emerging. We are told that, compared with the writer, the artist, the preacher, the jurist, the man of science is uncultivated; he finds it difficult to express himself; he has little standing in the general stream of cultural advance, and little common ground on which to stand with the arts man. William Henry Perkin is an example of the utter nonsense of this conception of the man of science. His insight into the economic possibilities of scientific discovery on the one hand, and into the contribution which the arts could make to the wellbeing of the working scientist on the other, make untenable any supposition that a difference need exist between the culture of the scientist and the culture of the arts man.

His interest, active and understanding, in the field of music and visual art, his practical bent in a number of directions, his preparedness to enter the field of finance, commerce, and industrial manufacture, his insight into ways of making money, his deeply religious convictions — all these are in themselves bigger things than any single incident that might happen in the course of his life. But it is these things, not the discovery of Mauve, that made him into the kind of man he was. That discovery was an incident — almost an accident. The exploitation of the discovery was in itself possible only because of his attributes as a whole man.

The world will for all time remember Perkin as the man who discovered the first synthetic dye. He should, however, be remembered as the kind of man to whom a single incident, such as that discovery, became important because he made it important. That kind of incident could happen to a great number of people without leading, as in his case, to the founding of great industries, or the material advancement of a science as a whole. The greatness of Perkin's contribution is that he was the kind of man who could make use of the things that happened to him.

The nonsense that is written about the alleged two cultures would not be written if there were not some underlying truth in the fact that the scientist in our present era tends to be a scientist and nothing more. We tend to regard the intellectual discipline of science and technology as being a complete and sufficient discipline for our appreciation of life around us — which it is not. We tend to believe that the currency of useful conversation is necessarily facts — which it is not. We tend to suspect that those amongst us who spend their leisure time from their laboratories or their factories in pursuit of the arts and the humanities are odd, and in any case are dispersing their energies unprofitably. In Perkin's day this was not so. Nor is it so today — for all of us. There are a great many more scientists with an insight into the arts and humanities than there are artists with a grasp of science. If we mention Albert Einstein, John Boyd Orr, Albert Schweitzer, James Jeans, and dozens more such musicians, philosophers, and writers — working scientists all — that is not to suggest that only the top men spread their cultural interests. I know few

* Reproduced in the December 1956 issue of the *Journal* (72, facing p. 557).

large scientific establishments and manufactories where there are not active drama, music, painting, and debating societies. I have personal experience of several groups of technical personnel at one level or another, who meet regularly for the purpose of domestic music-making, and also of painting and sketching groups in which there is as high a proportion of scientists as representatives of any other trade. Most of us will remember from student days university union debates at which the scientist generally had as much to say for himself, on anything and everything, as anybody. These activities are commonplace.

It is, however, true that we are tending more and more, from school upwards, to produce a kind of scientist who conforms to a pattern. We start to specialise in science when we are in our early teens. We omit to learn to speak the foreign languages our job makes it necessary to learn to read. We regard the learning of a musical instrument or enforced attendance at an Art Gallery as things which should be dropped before we get near to General Certificate "A" level. Too many of us suspect that any one who is paid to teach, and doesn't teach facts, is a charlatan, and we grow up with little appreciation of all the many sides of life that went to the making of a Perkin. And so it happens that writers with second-hand knowledge of our jobs, and no knowledge at all of our lives, have some grounds to pick on us.

I believe sincerely that if we allowed young people to grow up, spreading their interests as widely as possible, seeing to it that they lived against the background of many sides of human activity, which might include, as in Perkin's case, things as far apart as carpentry and chamber music, we should create the conditions for the emergence and survival of more men like Perkin. I believe this sufficiently strongly to say that, unless the training curricula of our schools and universities, and the attitude to life of us mature scientists and technologists, take account of these things, we shall steadily allow our heritage of outstanding geniuses to run to waste.

It has been said that great discoveries depend for their importance on being made by the right man at the right time and in the right place. What I have been saying is that Perkin was the right man. A person of much greater ability as an experimentalist and with a deeper knowledge as a scientist could easily have failed to make Perkin's discovery had he not also something of Perkin's make-up, the many facets of which I have touched on. That in itself is an important lesson which should teach us that an expert knowledge of chemistry and a high facility in experimentation do not of themselves make it likely that one will achieve discoveries.

So much for the right man. What of the right time and place? Perkin was born when the modern chemical age was at its beginning. It was not many years before his birth that Dalton had put forward that atomic theory which was to be the basis of chemical thought for over a century. This theory, propounding that atoms combine with each other in definite numbers, had led quickly to the possibility of giving formulae, in terms of numbers of different atoms, to known substances. Thus Perkin might know that quinine was $C_{20}H_{24}N_2O_2$ — and that is all he could know. It seemed possible, therefore, that a compound containing a known number of carbon, hydrogen, and nitrogen atoms might by oxidation be induced to take up a certain number of oxygen atoms and, if the numbers were chosen rightly, the end-product might be quinine. We know this to be entirely fallacious. But Perkin didn't. He knew just enough to make experiments on those lines appear worth while. Had he been born 50 years later, when the constitution of quinine had still not been worked out, or 100 years later, when it had, he could not have conceived that this experiment would lead him anywhere. To that extent he was born at the right time to make the experiment. A few years later pure aniline would have been available, and Perkin might not have obtained his Mauve dye, whose formation depended on the presence of *o*- and *p*-toluidines as impurities. Further, had he made his discovery a few years later his product would not have been the *first* highly coloured dye. It would have been just another dye. We could add to these time factors, but I think that the point is made.

As to place, one fact alone makes the point. Perkin could have gone to almost any school as a boy in England, and not heard of chemistry. It so happened that the City of London School was one of the very few schools where it was taught as a subject.

Thus we see Perkin, the right man at the right time in the right place. His personal greatness lay in his being the right man, and not in the accident of his birth, nor in the fact of his carrying out an experiment, which we know could not succeed, and turning it to his advantage. In fact, William Henry Perkin was great because he was what he was, and not because he made the discovery whose centenary we are honouring by our meeting tonight.

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(*MS. received 21st November 1956*)

Some Investigations into the Application of Dyes to Acrylic Fibres

B. KRAMRISCH

Meetings of the Huddersfield Section held jointly with the Huddersfield Section of the Royal Institute of Chemistry at Silvio's Café, Huddersfield, on 9th October 1956, Mr. J. Calvert in the chair; and of the Leeds Junior Branch, held at Leeds University on 27th November 1956, Dr. C. B. Stevens in the chair

The chief characteristics of polyacrylonitrile fibres are described briefly. Methods of application of disperse, basic (including Perkin's Mauve), Deorlene, acid, metal-complex, and chrome dyes to acrylic fibres, particularly Orlon 42 and Acrilan, are given in detail, and the main fastness properties of the dyeings are listed. Comparisons are made between the fastness properties, in particular to light and washing, of dyes on acrylic, natural, and other man-made fibres. Methods of dyeing unions of wool with Orlon 42 and Acrilan are detailed.

Ever increasing attention is being paid all over the world to the production, use, and processing of synthetic fibres. The three principal groups which have obtained a footing in the textile industry are the polyamide, polyester, and acrylic fibres. Each group of fibres has its own merits and demerits and has to find its own particular field of application in the textile industry.

Fibres of the acrylic group possess a number of attractive characteristics, such as soft handle, which makes them of interest for the ladies' knitwear trade, good all-round properties, which are of value in blends with wool, high resistance to degradation on exposure to sunlight, and good performance on washing.

A useful survey of the chemical and physical properties of synthetic fibres has been made by Koch [1], and a comparison of the main properties of these fibres is given in the "Synthetic Fibre Tables" issued from time to time by the *Textile World* [2]. A comparison of the resistance to weathering of acrylic and other fibres has been made by Rein [3]. Table I gives a list of acrylic fibres, but is not necessarily comprehensive and may include fibres which are not yet produced on a commercial scale.

Acrylic fibres may contain at least 85% of polyacrylonitrile, derived as follows—

$$\underset{\text{Acetylene}}{CH{:}CH} + \underset{\substack{\text{Hydrogen}\\\text{cyanide}}}{HCN} \rightarrow \underset{\substack{\text{Acrylonitrile}\\\text{(vinyl cyanide)}}}{CH_2{:}CH{\cdot}CN} \rightarrow \underset{\text{Polyacrylonitrile}}{[CH_2{\cdot}CH(CN)]_n}$$

Some acrylic fibres are copolymers of acrylonitrile, and it is the introduction of certain derivatives which apparently confers on acrylic fibres their affinity for wool dyes, as is shown, for example, with Acrilan. A wide variety of vinyl and other compounds have been mentioned in the technical and patent literature in this connection, including the following—2-vinylpyridine, alkyl-vinylpyridines, vinyl acetate, vinylglyoxaline, *N*-vinyl-lactams, vinyl ethers of aminoalcohols, 1-vinylglyoxaline and an ethylenic compound (e.g. vinyl acetate, methacrylonitrile, or vinyl chloride), *N*-benzylacrylamides, maleic monohydrazide, phenol–aldehyde condensates, nicotinamides, *iso*-propyl acetate–low-alkyl acrylate copolymers, amine derivatives of vinyl esters of halogenated monocarboxylic acids, and esters of acrylic acid and methacrylic acid.

The general theme of this paper is to outline the application and properties of various classes of dyes on acrylic fibres and also to describe the dyeing of unions of these fibres with wool.

In order to form a better picture of the behaviour of dyes on acrylic fibres a number of comparisons are made between the application and fastness properties of dyes on these fibres and on other synthetic and man-made fibres such as nylon, Terylene, secondary cellulose acetate, and cellulose triacetate, and also three natural fibres—wool, cotton, and silk.

The main references will be made to Orlon 42 and Acrilan. As far as the former is concerned, however, the remarks can be taken as a general

TABLE I

Acrylic Fibres

Commercial Name	Previous Designation	Manufacturers	Address
Acrilan	—	Chemstrand Corpn.	Decatur, Alabama, U.S.A.
Courtelle	—	Courtaulds Ltd.	Coventry, England
Creslan	X54	American Cyanamid Co.	Stamford, Conn., U.S.A.
Crylor	Fibre D	Société Rhodiacéta	Lyons, France
Dolan	K8F Kelheim	Süddeutsche Zellwolle AG.	Kelheim, Germany
Dralon	Bayer acrylic fibre	Farbenfabriken Bayer AG.	Dormagen, Germany
Dynel	—	Carbide & Carbon Chemicals Co.	New York, U.S.A.
Nitrilon	—	Kirov Textile Institute	Leningrad, U.S.S.R.
Nymcrylon	53	N.V. Kunstzijdespinnerij	Nijmegen, Holland
Orlon 81 and 42	Fibre A	E.I. du Pont de Nemours & Co. Inc.	Wilmington, Del., U.S.A.
PAN	—	Cassella Farbwerke Mainkur AG.	Frankfurt-am-Main, Germany
Redon	PX II	Phrix-Werke AG.	Hamburg, Germany
Sinsten	—	—	Japan
Verel	—	Tennessee Eastman Corpn.	Kingsport, Tenn., U.S.A.
Wolcrylon	—	VEB Film- und Chemiefaserwerke Agfa	Wolfen, Kreis Bitterfeld, Germany
X-51	—	American Cyanamid Co.	Stamford, Conn., U.S.A.
Zefran	—	Dow Chemical Co.	Midland, Mich., U.S.A.

guide to the behaviour of dyes on American and Continental acrylic fibres akin to Orlon 42. Nevertheless, certain differences in such properties as rate of dyeing, affinity, fastness, and shade are likely to be encountered.

Orlon 81, which is the filament form of Orlon, has only limited use in the textile industry. With normal dyeing and printing techniques it is possible to produce only pale colours on Orlon 81, and high-temperature dyeing is generally necessary to obtain medium to full depths. With this in mind, this paper deals only with the staple variety— Orlon 42.

A number of papers have been published on the dyeing of acrylic fibres and unions of these fibres with other materials. For instance, Stern [4] and Walls [5] have dealt with Orlon 42, and Woodruff [6] and Hindle [7] with Acrilan.

DISPERSE DYES

Disperse dyes have found a place in the dyeing and printing of practically every type of synthetic fibre and have served as a ready means of producing a wide range of colours. The fastness properties of the coloured material have been found adequate for a diversity of textile materials, although in certain cases either the colour or the fastness obtainable falls short of the standard required, and as a result consideration has had to be given to other groups of dyes.

The use of disperse dyes on acrylic fibres is limited to a certain extent, as some show relatively low saturation values. An interesting feature of dyes on these fibres is that the shades produced with the orange, scarlet, red, maroon, and violet members of the ranges are reasonably comparable with those obtained on secondary cellulose acetate and cellulose triacetate, in contrast with nylon and other polyamide fibres, where noticeably bluer and duller colours are produced with many disperse dyes.

A comparative study of the temperature-range properties of disperse dyes on various fibres shows some interesting features. This behaviour is illustrated by tests with 2% Cibacet Sapphire Blue 4G (Fig. 1) and 2% Cibacet Orange 4R (Fig. 2). These two dyes were applied for 90 min. at temperatures ranging from 20°C. up to 120°C., and the depth of colour obtained was assessed visually by comparison with ranges of dyeings on secondary cellulose acetate. In all cases dyeings were prepared in a 50:1 liquor with no additions of assistants to the dyebath. The following materials were employed—

Secondary cellulose acetate— 22s Celafibre (BrC) yarn
Cellulose triacetate— 12s Courpleta (Courtaulds) yarn
Terylene (ICI)— 2/28s yarn
Acrilan (Chemstrand)— 18s yarn
Nylon (British Nylon Spinners Ltd.)— yarn
Orlon 42 (DuP)— regular 2/26s yarn

all in staple form.

Several facts can be deduced from these graphs—

(1) The uptake of dye at lower temperatures is much less with Terylene (dyed without carrier) than with cellulose triacetate, and this in turn is below that of secondary cellulose acetate, which

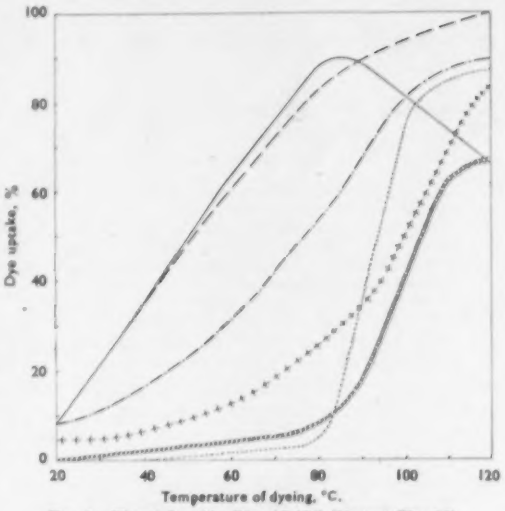

FIG. 1— Cibacet Sapphire Blue 4G (C.I. Disperse Blue 16)

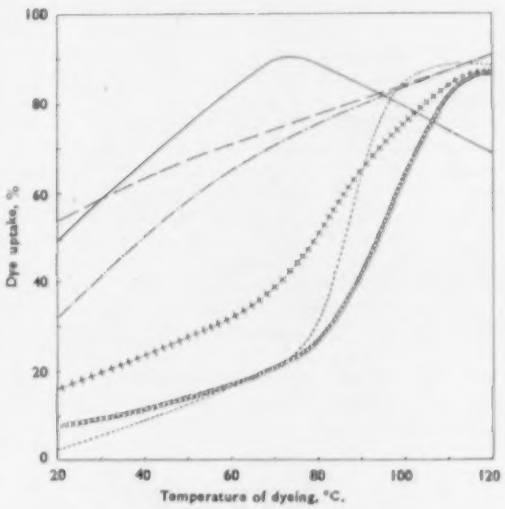

FIG. 2— Cibacet Orange 4R (C.I. Disperse Orange 8)

Cellulose acetate
Nylon
Cellulose triacetate
Terylene
Orlon 42
Acrilan

FIG. 1 and 2— Temperature-range Properties of Disperse Dyes on Various Fibres

confirms the general experience gained with disperse dyes on these three types of fibre. With acrylic fibres, the rate of uptake at lower temperatures is even less than with the other fibres mentioned.

(2) With one of the dyes examined — Cibacet Sapphire Blue 4G — the temperature-range properties of Orlon 42 and Acrilan are very similar, showing a marked increase in dye-uptake in the region of 80°C., which indicates the need for care in bulk application. With Cibacet Orange 4R a similar type of change in dyeing behaviour in the 70°C. region is apparent.

(3) The phenomenon mentioned under (2) above is not apparent with either dye on secondary

TABLE II

Substrate	Depth (%)	Fastness to Light [8]	Fastness to Washing [9] Change in Colour	Staining of Wool	Staining of Cotton	Staining of Secondary Cellulose Acetate
CIBACET ORANGE 4R (C.I. Disperse Orange 8)						
Secondary cellulose acetate ...	0·7	4	2	4	4–5	3
Cellulose triacetate	1·0	5	4–5	4–5	4–5	3
Nylon	0·6	4	3	3–4	4–5	2–3
Terylene	1·0	5–6	5	5	5	4–5
Orlon 42	1·0	5–6	4–5	5	5	4
Acrilan	0·5	5	4–5	5	5	4
CIBACET SAPPHIRE BLUE 4G (C.I. Disperse Blue 16)						
Secondary cellulose acetate ...	0·7	4	3–4	4	4–5	3
Cellulose triacetate	0·7	3	4–5	4–5	5	4
Nylon	0·7	5–6	3–4	4	4	2–3
Terylene	1·0	4–5	3–4	4	4–5	3
Orlon 42	1·0	6	4	4–5	5	3–4
Acrilan	0·7	5–6	4–5	5	5	3–4

cellulose acetate and cellulose triacetate, which give much smoother curves.

The above dyes were then compared for fastness to light and washing on the same series of fibres. In the case of the light-fastness tests daylight exposure was made against the British wool standards[8], and for fastness to washing the S.D.C. No. 2 test[9] was employed. Dyeings of similar colour value were prepared, and the results are given in Table II.

The main feature of these results is the high standard of fastness to light and washing achieved on acrylic fibres and the advantages in this regard over some of the other fibres, especially secondary cellulose acetate.

Many disperse dyes, particularly blues, show gas-fume fading on both cellulose acetates. They are, however, free from this defect on polyamide, polyester, and acrylic fibres.

Although disperse dyes as a group are of good fastness to dry-heat pleating on acrylic fibres, and in this respect are generally superior to the same dyes on polyamide fibres, they are not all suitable for materials which have to be steam-pleated, as some tend to sublime and mark-off, especially when dyed in full colours. This is a point to be watched when acrylic fibres are blended with wool for materials which are to be steam-pleated.

BASIC DYES

The interesting observation has been made that basic dyes as a class show good affinity for acrylic fibres under slightly acidic dyeing conditions. A range of bright full colours of very high fastness to washing and even alkaline milling are obtained, and the fastness to light is significantly better than that of the same dyes on tannin-mordanted cotton. As a result, attention has again been focussed on a group of dyes which for many years has had only relatively limited application in the textile industry.

A suggested process for basic dyes on Orlon 42 is to dye with the addition of 1 ml. of acetic acid (40%) and 0·5 ml. of Neolan Salt P per litre. Dyeing is begun at 40–50°C. and the liquor raised slowly to the boil, with special care at 90–95°C. This region is very critical, for the rate of strike increases very markedly, and as a result unlevel results may be produced unless sufficient care is exercised. Dyeing is completed after boiling for 1 hr.

In the case of Acrilan it is suggested that dyeing be carried out with the addition of 5% ammonium sulphate and 0·5–1·0 ml. Neolan Salt P per litre. Again dyeing is begun at 40–50°C., the liquor raised gradually to the boil, and boiling continued for 1 hr. On the whole Acrilan does not appear to be quite as sensitive to temperature of dyeing as is Orlon 42.

In general, basic dyes are not as productive on Acrilan as on Orlon 42.

Two typical basic dyes, Magenta and Rhodamine B, were compared for fastness to light (daylight exposure[8]) and washing (S.D.C. Mechanical Washing Test A[9]) on Orlon 42, Acrilan, tannin-mordanted cotton, silk, and cellulose acetate. The results (Table III) illustrate the improved fastness to light and washing of basic dyes on acrylic fibres compared with natural fibres.

TABLE III

Substrate	Depth (%)	Fastness to Light [8]	Fastness to Washing (S.D.C. Mechanical Washing Test A[9]) Change in Colour	Staining of Wool	Staining of Cotton
MAGENTA (C.I. Basic Violet 14)					
Orlon 42 ...	0·15	3–4	5	5	5
Acrilan ...	0·6	3	4	4	5
Cotton	0·2	1	4	2	3
Silk	0·15	2	1	2	2
Cellulose acetate	0·15	1	4	3–4	4–5
RHODAMINE B (C.I. Basic Violet 10)					
Orlon 42 ...	1·5	3–4	4	5	5
Acrilan ...	6·0	2–3	3–4	4–5	4–5
Cotton	1·5	1	2	1	2
Silk	1·0	3	2	3	2
Cellulose acetate	1·5	4	2	4–5	4–5

TABLE IV

Fastness of Dyeings of Mauve (C.I. 50245)

Substrate	Depth (%)	Fastness to Light [8]	Fastness to Washing (S.D.C. No. 2 Test [9]) Staining of				
		Change in Colour	Orlon 42	Acrilan	Cotton	Cellulose Acetate	Wool
Orlon 42	0·6	3	5	5	—	—	—	5
Acrilan	0·6	2–3	5	—	5	—	—	4–5
Cotton	0·5	2	3–4	—	—	4	—	4
Silk	0·4	2–3	3–4	—	—	4	—	4–5
Cellulose acetate ...	0·4	1	5	—	—	—	5	5

In view of the current interest in the Perkin Centenary, it was thought worth while carrying out tests with Mauve, a basic dye. Dyeings have been prepared at comparable strengths and assessed for light fastness by daylight exposure [8] and for washing fastness by the S.D.C. No. 2 Test [9]. It will be seen (Table IV) that Mauve shows a slight improvement in fastness to light and washing on acrylic fibres compared with natural fibres.

DEORLENE DYES

In view of the interesting results obtained with basic dyes on acrylic fibres, further attention has been paid to this class, and the Deorlenes, which are modified basic dyes, have been developed. One of the objects of developing this group of dyes has been to extend the colour range and at the same time give better fastness to light than that obtainable with ordinary basic dyes, while maintaining the brightness and high fastness to washing.

All members of the Deorlene range are applicable to Orlon 42, and apart from the navy blue and black are applied in a similar manner to basic dyes. The following procedure is recommended for the navy and the black—

Set the dyebath with (per litre)—

1 ml.	Acetic acid (40%)
1–2 ml.	Formaldehyde (40%)
0·5 ml.	Neolan Salt P

Commence at 50°c., raise slowly to the boil, and maintain for 1½–2 hr. at the boil. These particulars refer to a 40–50:1 liquor ratio. In short liquors, the acetic acid should be increased from 1 to 4 ml. and the higher amount of formaldehyde used.

The Deorlenes are applied to Acrilan in a similar manner to basic dyes, and apart from the navy and the black, all members of the range are of value. The colour value is in general not as good on Acrilan as on Orlon 42.

An interesting dye is Deorlene Blue E5G, which on Orlon 42 and Acrilan gives bright turquoise blues of good fastness to washing and light. The light fastness is of the order of 5 on both materials and compares favourably with a light fastness of 2–3 of similar colours on wool normally produced with triphenylmethane acid dyes.

A comparison of the fastness to light (daylight exposure [8]) and washing (S.D.C. Mechanical Washing Test A [9]) of Deorlene Brilliant Red 3B on various substrates (Table V) illustrates the superior fastness of a Deorlene dye on acrylic fibres compared with other substrates.

TABLE V

Fastness of Deorlene Brilliant Red 3B on Various Substrates

Substrate	Depth (%)	Fastness to Light [8]	Fastness to Washing (S.D.C. Mechanical Washing Test A [9])		
			Change in Colour	Staining of Wool	Staining of Cotton
Orlon 42 ...	0·25	4	4	5	5
Acrilan	1·0	3–4	4	4	4–5
Cotton	0·5	1	3	2	3
Silk	0·35	2–3	1	2	3
Cellulose acetate	0·25	1	3	3	4–5

In general, basic and Deorlene dyes on both Orlon 42 and Acrilan show good fastness to both dry-heat pleating and steam pleating.

ACID DYES

Kiton, Alizarine, and Benzyl dyes have virtually no affinity for Orlon 42 if an attempt is made to apply them by conventional wool-dyeing techniques, but quite a number can be applied by the cuprous-ion [10] and allied processes. In general, monosulphonated dyes show better productivity than dyes of higher degrees of sulphonation. Processes of this type have been limited in bulk application by difficulties in pattern matching and reproduction of shades. This position is confirmed by published work by Walls [5] and others.

Most dyes of this group are applicable to Acrilan, but lower pH values are needed than for the application of the same dyes to wool. For instance, dyes of the Benzyl and Alizarine types are normally applied to wool in the presence either of an organic acid or of an ammonium salt of an organic or a mineral acid. A mineral acid (sulphuric acid) is needed for dyeing Acrilan with the same dyes to obtain adequate depth of colour. The acid levelling type of dye, which is normally applied to wool in the presence of 2% sulphuric acid, requires a further 6% sulphuric acid to produce a sufficient depth of colour.

Further improvement in depth can often be achieved by pretreatment of the Acrilan with 2 g. of sulphuric acid per litre at the boil for 30 min., followed by a rinse and dyeing in the presence of 4% Cibalan Salt S, if necessary with the addition of a little sulphuric acid towards the end of the dyeing operation.

An interesting illustration of this method is Benzyl Fast Red 2RL, which shows very little affinity for Acrilan when applied from a sulphuric-acid dyebath. If the Acrilan is pretreated with

sulphuric acid as indicated above, and, after rinsing, is dyed with the aid of 4% Cibalan Salt S only, a full scarlet of good wash fastness is produced.

Acid dyes differ in fastness to light and washing on acrylic fibres and on wool. In some cases unexpected results are obtained, particularly with respect to light fastness, and therefore prior tests are always advisable.

METAL-COMPLEX DYES

Dyes of this type are of no value for Orlon 42. The 1 : 1 chromium complexes (Neolans) are applicable to Acrilan in a similar manner to wool, namely with 8% sulphuric acid or 5% sulphuric acid and 2% Neolan Salt P at the boil for 90 min., and they build up to quite full colours. The foregoing quantities relate to dyeings prepared in 40–50 : 1 liquors, and corresponding adjustments are necessary when liquor ratios deviate to any marked degree from these values. Reduction in the amount of sulphuric acid and replacement by Neolan Salt P affects neither the colour obtained nor the resulting fastness properties.

Neolan dyes show very little affinity for Acrilan when applied in the presence of organic acids. Dyeings prepared with sulphuric acid as indicated above possess good fastness to washing and alkaline milling, and in general the results are better than with the same dyes on wool. With Neolan Blacks WA extra and 2G, maximum wet fastness is obtained by adding 0·3–0·5% sodium or potassium dichromate after one hour's dyeing at the boil in a sulphuric-acid bath.

Neolan Black WA extra gives a reddish black and Neolan Black 2G a dull green, and attractive blacks are obtained by combining the two dyes. The colour is fuller than that obtainable with a chrome black, and although the fastness to washing and alkaline milling of the Neolan Blacks on Acrilan is better than on wool, it is not quite up to that obtained with a chrome black, with respect to staining of wool and cotton.

It is interesting to note that Neolans developed primarily for leather dyeing, such as Neolan Light Brown C, Neolan Orange 2GC (C.I. Acid Orange 61), and Neolan Brown TCN, show good productivity and fastness to light and washing on Acrilan. Neolan Brown TCN is of special interest for medium to dark browns and shows a light fastness of 5 on Acrilan, which is about the average for the majority of the Neolan range on this fibre.

A comparison of the temperature-range properties of three Neolan dyes on wool and Acrilan (Fig. 3) indicates greater care in the application of these dyes to Acrilan than to wool in the region from 60°C. up to the boil.

Many 2 : 1 complexes of dye and metal (chromium or cobalt), i.e. Cibalans, are applicable to Acrilan with the addition of 8% sulphuric acid for 90 min. at the boil. This recommendation relates to dyeings carried out in 40–50 : 1 liquors, and correspondingly decreased amounts of acid are advisable in shorter liquors.

Very low productivity is obtained when dyes of this type are applied by the normal procedure used

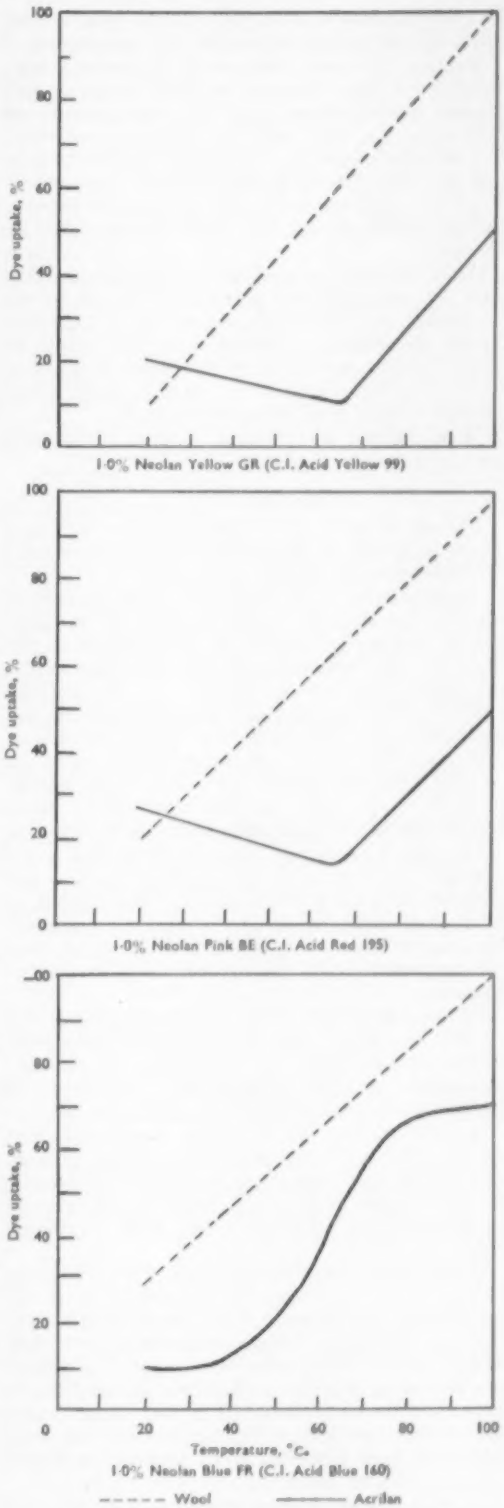

FIG. 3—Temperature-range Properties of Neolan Dyes on Wool and Acrilan dyed separately

for wool, namely with the aid of ammonium sulphate, and apparently a low pH is essential.

Dyeings produced with sulphuric acid as indicated have good fastness to light, washing, and alkaline and acid milling. The productivity is noticeably less than with the same dyes on wool, and therefore their use is confined chiefly to pale colours. The use of 8% formic acid in place of 8% sulphuric acid tends to give dyeings of inferior milling fastness, and the levelness may also be inferior.

An alternative method is to pretreat the Acrilan with 2 g. sulphuric acid per litre for 30 min. at the boil, rinse, and dye in the presence of 4% Cibalan Salt S, if necessary followed by a little sulphuric acid. Dyeings produced in this manner compare in colour and fastness to washing with those applied from a sulphuric-acid dyebath.

Most Neolan and Cibalan dyeings on Acrilan are fast to steam pleating.

CHROME DYES

Chrome dyes are of virtually no value for Orlon 42, as they will not dye this fibre by either normal or modified wool-dyeing techniques.

Many members of the range are applicable to Acrilan but require a relatively low pH in application, increased amounts of acid and dichromate, and also an extension of the chroming period compared with wool dyeing to develop the chromium lake satisfactorily. In this respect similar difficulties are encountered on polyamide fibres, although chelation is generally more rapid on Acrilan than on polyamide fibres. The results, however, vary with the individual dye. Interesting examples of these differences are as follows—

CHROME FAST BLUE 2R (C.I. Acid Red 14)—This dye gives a red in the unchromed state and chromes to a navy blue on wool. On Acrilan an intensified chroming process is required to achieve this result, whereas on nylon lake formation does not appear to take place, even with intensified chroming.

CHROME FAST GREEN G (C.I. Mordant Green 17) — This dye gives a violet in the unchromed state and a green on both wool and Acrilan by a normal chroming process. Intensified chroming is necessary on nylon to produce a similar effect.

As a class, chrome dyes give dyeings of good fastness to light, washing, and alkaline milling, and on the whole they compare with wool dyeings in this regard. Some differences in wet-fastness properties may occur, however, with individual dyes.

Most chrome dyes give dyeings of good fastness to steam pleating. They are of special interest for full colours, such as navy, bottle green, dark brown, maroon, and black. Chrome Fast Black PV (C.I. Mordant Black 9) is to be recommended as a basis for blacks; as it gives a slightly "plummy" tone, it is advisable to shade it with Chrome Fast Yellow O (C.I. Mordant Yellow 34) and possibly also some Alizarine Fast Green G (C.I. Acid Green 25) to achieve a better black.

A suggested application procedure is to dye with 3% sulphuric acid, followed by a further 3% sulphuric acid, dyeing for 1 hr. at the boil altogether, wash off, chrome in a fresh bath with 5% sodium or potassium dichromate and 3% sulphuric acid for 1 hr. at the boil, and follow with a final washing off. Dyeings produced on these lines exhibit high fastness to light, washing, alkaline milling, and steam pleating.

VAT DYES

Mention is made in the technical literature of the application of vat dyes and esters of leuco vat dyes to acrylic fibres, and in some instances modification of conventional dyeing processes may be required. It is questionable, however, whether these types of dyes will find wide application on acrylic fibres, as a wide range of colours with adequate fastness for most requirements is obtainable with other classes of dyes.

UNIONS OF ORLON 42 AND WOOL

Unions of Orlon 42 and wool can be dyed with disperse (Cibacet) or Deorlene dyes, any staining of the wool removed by clearing with potassium permanganate and acetic acid or formic acid and Hydrosulphite BZ water-soluble (Ciba), and the wool then cross-dyed with Cibalan, Benzyl, or Alizarine dyes from a neutral dyebath or in the presence of ammonium acetate or ammonium sulphate. Disperse dyes are generally preferable for pastel colours.

Clearing of the stained wool is essential to ensure the production of dyeings of maximum fastness.

UNIONS OF ACRILAN AND WOOL

(*a*) SOLID SHADES

It is difficult to generalise regarding the production of solid shades on unions of Acrilan and wool, as each colour needs individual consideration regarding selection of dyes. The position can, however, be summarised under the following five groupings—

(*1*) *Combinations of Disperse* (*Cibacet*) *and Wool Dyes*

Combinations of disperse (Cibacet) dyes for the Acrilan portion and neutral-dyeing wool (Benzyl, Alizarine, or Cibalan) dyes for the wool portion can be applied by a one-bath process at the boil. The wool, however, is stained by the disperse dye, and this staining must be removed; otherwise, the fastness of the wool portion to light, rubbing, and wet treatments will be reduced to an appreciable degree. Although this staining could be removed partially or completely by a hydrosulphite treatment, not all wool dyes will withstand a treatment of this nature.

With this in mind, it is often preferable to resort to a two-bath process, viz. to dye the Acrilan first with disperse dyes, clear the stained wool with hydrosulphite, and then cross-dye the cleared wool from a fresh bath with acid, Benzyl, Alizarine, or Cibalan dyes by conventional dyeing methods, possibly by dyeing at a slightly reduced temperature. This procedure is of special interest for mode shades, although if the material is to be steam-pleated, the fastness of the disperse dye to this process should be checked.

(2) *Acid Dyes*

When acid dyes are applied by normal wool-dyeing techniques, preferential dyeing of the wool portion of the union occurs. By lowering the pH of application, viz. by starting with 2% sulphuric acid and following with a further 6% sulphuric acid, dyeing for 60–90 min. at the boil altogether, this difference is reduced considerably, and many acid dyes give good solidity on the union. The results, however, may vary with the individual dye, and also the Acrilan portion of the union may be of lower light fastness than the wool portion.

(3) *Metal-complex (Neolan) Dyes*

Applied by the normal wool-dyeing technique, namely with 8% sulphuric acid for 90 min. at the boil, many Neolans give quite good solidity over a range of light, medium, and full colours. A good boil is, however, essential, otherwise the Acrilan portion of the union remains lighter.

In many cases pretreatment of the union with 2 g. sulphuric acid per litre for 30 min. at the boil, followed by a rinse and dyeing from a sulphuric-acid dyebath, or with Neolan Salt P followed by sulphuric acid, may give further improvement in solidity.

Care in selection of dyes is advisable, both for production of solid shades and for comparable fastness to light on the two fibres. The following Neolans are of special interest—

Light Brown C
Brown TCN
Bordeaux BE (C.I. Acid Red 212)
Dark Blue F2R
Navy Blue 2RLC (for navies)
Black WA extra and 2G (C.I. Acid Black 52 and 54) (for blacks)

(4) *Metal-complex (Cibalan) Dyes*

When applied by the normal wool-dyeing technique, viz. with 2% ammonium sulphate for 1 hr. at the boil, preferential dyeing of the wool component takes place, and the Acrilan is appreciably lighter in colour than the wool.

A range of solid shades can be obtained by pretreatment of the union with 2 g. sulphuric acid per litre for 30 min. at the boil followed by a rinse and dyeing from a fresh bath with the addition of 4% Cibalan Salt S for 90 min. at the boil. 1-2% Cibalan Salt N may also be added. This procedure is confined to pale colours, and care in dye selection is necessary. Combinations of the following Cibalan dyes are mainly recommended—

Yellow GRL (C.I. Acid Yellow 116) (+ FGL in certain cases)
Red 2GL (C.I. Acid Red 211)
Blue BL (C.I. Acid Blue 168)

(5) *Combinations of Modified Basic (Deorlene) and Wool Dyes*

A two-bath technique is necessary, the Acrilan being dyed first with Deorlene dyes with the addition of 2% ammonium sulphate for 1 hr. at the boil. Care is advisable in order to obtain levelness on the Acrilan. The wool portion of the union is usually stained to an appreciable degree and will possess only "basic colour fastness", so that a clearing treatment with hydrosulphite is essential. After washing off, the cleared wool can be cross-dyed in a fresh bath with acid, Benzyl, or Cibalan dyes by conventional wool-dyeing techniques.

(b) TONE-IN-TONE AND INGRAIN EFFECTS

These can be produced with acid, Cibalan, or chrome dyes applied by normal wool-dyeing methods. The Acrilan portion of the union is usually dyed paler than the wool, and where a high standard of light fastness of the finished goods is desirable, a preliminary check is advisable.

* * *

I acknowledge my indebtedness to the Clayton Dyestuffs Co. Ltd. for permission to publish this paper and to Mr. A. K. Taylor and Mr. W. B. Harrison for assistance with the practical work.

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(*MS. received 9th November 1956*)

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Discussion

(at Huddersfield)

Mr. B. WAGSTAFFE: Is there any method for applying Cibalan Black BGL to Acrilan?

Mr. KRAMRISCH: Depending on the pH of the dyebath, Cibalan Black BGL gives only grey to "plummy" shades on Acrilan when applied at temperatures up to the boil. A better black is obtainable, however, at 120°C.

Mr. A. E. BROSCOMBE: Can any reason be given for the shade differences noted on wool and Acrilan when unions of these fibres are dyed with Cibalan dyes?

Mr. KRAMRISCH: Firstly the two fibres differ in constitution and secondly the pH of dyeing differs from that normally used for wool.

Mr. S. L. PEEL: How do Cibalan dyes compare for fastness to alkaline milling on Acrilan and wool?

Mr. KRAMRISCH: When applied with 8% sulphuric acid, the Cibalans as a range give dyeings on Acrilan of fastness to alkaline milling comparable with that of similar dyeings on wool produced by the usual techniques. When 8% formic acid is employed, the milling fastness tends to be not quite as good, although this can vary with the individual dye.

Mr. W. BEAL: Difficulties are likely to occur when dyeing Acrilan owing to setting of the

material if cooling is too rapid. Are there any means of overcoming this trouble?

Mr. KRAMRISCH: This behaviour is due to the physical properties of the fibre, and only careful control in bulk will give the best results. A similar position occurs with Orlon 42.

Mr. BEAL: Anomalous fading has been observed with certain combinations of disperse dyes on Acrilan, the fastness of a mixture dyeing being significantly below that expected from the fastness to light of the individual components. Has a similar effect been observed with combinations of wool dyes on Acrilan?

Mr. KRAMRISCH: The phenomenon reported by Mr. Beal is akin to that observed with individual combinations of yellow and blue disperse dyes on cellulose triacetate under some exposure conditions. As far as wool dyes are concerned, the very large number applicable to Acrilan makes it impossible at this stage to give a complete statement. It can be said, however, that none of the combinations tested so far has given unexpected results.

Mr. BALMFORTH: What is the maximum amount of sulphuric acid needed for dyeing Acrilan?

Mr. KRAMRISCH: 8% Sulphuric acid calculated on the weight of Acrilan is the maximum amount suggested for applying metal-complex dyes. This quantity relates to dyeings carried out in liquor ratios of 40–50 : 1, and in shorter liquors it can be reduced somewhat. Moreover, the amount of acid can be reduced by using a proportion of Neolan Salt P.

Mr. BALMFORTH: What are suggested stripping processes for Deorlene dyes on Orlon 42?

Mr. KRAMRISCH: Two recommended methods are as follows—

(*a*) 2 g. ... Sodium chlorite; 2 ml.... Formic acid (85%); 1 ml.... Claytavon B } 1 hr. at the boil

(*b*) 2·0 g. ... Sodium chlorite; 1·5 g. ... Disodium phosphate; 1·5 ml.... Nitric acid (conc.); 1·0 ml.... Claytavon B } 1 hr. at the boil

Stainless-steel vessels are to be avoided for this type of treatment.

COMMUNICATIONS

Measurement of Diffusion Coefficients of Azoic Coupling Components (2-Hydroxy-3-naphthanilides) in Cellulose

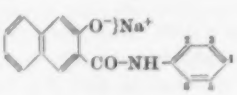
A. S. DUNN

Though several authors have previously studied the equilibrium sorption properties of the anilides of 2-hydroxy-3-naphthoic acid, no measurements of the diffusion coefficients of these molecules in cellulose have been made. A suitable apparatus is described for measuring the rapid rates of dyeing which occur, using thick cellulose model filaments. Diffusion coefficients and energies of activation for diffusion of six coupling components have been evaluated.

Most of the substantive azoic coupling components are substituted anilides of 2-hydroxy-3-naphthoic acid (I). Their affinities ($-\Delta G^\circ$) for cellulose[1] are considerably lower than those of direct dyes, being of the order of 3 kcal./mole, whereas values of $-\Delta G^\circ$ for direct dyes[2] under similar conditions lie in the range 5–8 kcal./mole. The rate of diffusion of these compounds in cellulose is much greater than that of direct dyes; this, and the greater difficulty of measuring the amount of coupling component on the fibre, appear to have prevented quantitative measurement of their diffusion coefficients hitherto. These difficulties, however, have now been overcome.

EXPERIMENTAL

The coupling components used all had the general formula I with the substituents indicated in Table I.



(I)

Solutions were prepared by dissolving the coupling component by heating in sufficient sodium hydroxide solution to give the stated

TABLE I

Properties of Coupling Components

Brenthol (ICI)	Substituents 2	3	4	5	Recryst. from	M.p. (corr.) (°C.)	Absorption Max. (mμ.)	Molar Extinction Coefficient*
AS	—	—	—	—	96% Ethanol	255·5	403	3800
OT	CH_3	—	—	—	96% Ethanol	200·5	402	3680
FR	CH_3O	—	—	—	Glacial acetic acid	168·5	408	3940
PA	—	—	CH_3O	—	Glacial acetic acid	239	402	4100
FO	CH_3O	—	—	CH_3O	60% Ethanol	187	402	3920
MN	—	NO_2	—	—	Glacial acetic acid	224	406	3020
AN			—	—	Glacial acetic acid	249	406	3590

* Of the sodium salt in 25% (vol./vol.) pyridine–water containing 0·025 N. sodium hydroxide.

excess. Solutions darken on standing, and so they were used as soon after preparation as possible.

Model filaments were prepared by extruding viscose into 10% ammonium sulphate and subsequently regenerating with sulphuric acid. These filaments were of circular cross-section and had a diameter of about 0·06 cm. after drying out and reswelling: the degree of swelling (i.e. weight swollen/weight dry) of such isotropic filaments is about 1·9.

Under the conditions used, the greater part of the equilibrium absorption of coupling component is taken up in the first 15 min. Since it is essential in measuring diffusion coefficients to maintain a rate of stirring sufficiently fast to ensure that the rate of uptake does not become controlled by diffusion in the liquid phase, sampling techniques requiring interruption of the experiment are unsuitable. The apparatus (shown in Fig. 1) was devised to provide continuous sampling by withdrawing the filament from the dyebath at constant speed. To start the experiment the model filament *A*, preswollen in water, is inserted into the dyebath *B* by moving the sliding pulley *C* from position *C'*: this operation may be completed within 3 sec. A 1-g. weight *H* serves to keep the filament taut.

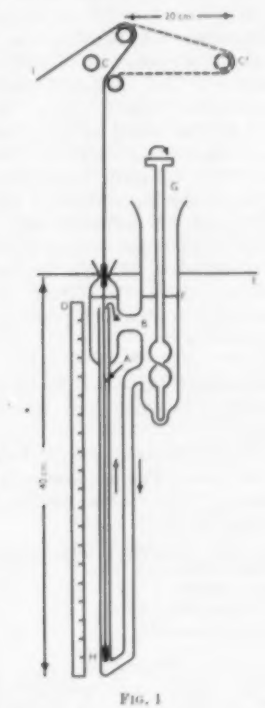


FIG. 1

The wet length of the filament is read on the scale *D*. The winding motor is started; by means of suitable gearing it withdraws the filament from the bath at a rate of 10 cm./min. The position of the weight *H* on the end of the filament is read periodically. When the filament has been completely withdrawn it is allowed to dry out, and the dry length of the impregnated portion is measured. This portion is cut into at least five equal lengths and a top portion. The light yellow colour imparted by the coupling components does not reveal the beginning of the impregnation clearly. The colour of the top portion is therefore developed with a suitable base (Brentamine Fast Corinth V Salt (ICI) was used to give a distinct brown colour): the dry length of the impregnated portion of the filament can then be obtained. The coupling component is extracted from the rest of the samples with 25% (vol./vol.) pyridine containing 0·025 N. sodium hydroxide and determined spectrophotometrically with the aid of the calibration data in Table I. Beer's law was found to be followed at the concentrations used (~10^{-5} M.). The filament samples are then dried and weighed.

From the scale readings a graph of length of filament withdrawn against time is constructed. The filament dries as it is withdrawn, so that the wet length of each specimen must be obtained by multiplication of the dry length by the ratio of the wet and dry lengths of the "dyed" portion. From the graph the mean time of immersion of each specimen is read off. Graphs of "dye" uptake against time can then be constructed.

The equilibrium absorption of coupling components at the relevant liquor concentrations were determined in separate experiments.

At fractional saturations (c_t/c_∞) less than 0·05 Hill's equation for diffusion into an infinite cylinder [3] reduces to the simple form —

$$\frac{c_t}{c_\infty} = \left(\frac{16Dt}{\pi r^2}\right)^{\frac{1}{2}}$$

where c_t is the dye concentration at time t, c_∞ the equilibrium uptake, D the diffusion coefficient, and r the radius of the filament. At $c_t/c_\infty = 0{\cdot}2$ the simple form and the full equation differ by only 4%, which is likely to be inappreciable experimentally: in practice, linear plots of c_t/c_∞ against $t^{\frac{1}{2}}$ were usually obtained, so that the simple form could be used to evaluate diffusion coefficients; otherwise, the diffusion coefficient could be calculated from each point from values of Dt/r^2 corresponding to c_t/c_∞ read off from a theoretical graph of c_t/c_∞ against Dt/r^2 constructed from Hill's equation [3, 4].

RESULTS

A stirring speed of 700 r.p.m. was normally used. Table II shows that this was amply sufficient to ensure that diffusion in the fibre should be the rate-controlling process.

TABLE II

Effect of Rate of Stirring

(0·005 M. Brenthol AS in presence of 0·025 N. excess NaOH at 25°C. $c_\infty = 5{\cdot}05 \times 10^{-3}$ mole/kg.)

Rate of stirring, r.p.m.	700	500	250	125
$10^6 D$, cm.²/sec.	3·89, 3·66	3·38, 3·29	3·84, 3·98	2·64, 3·38
Mean	3·78	3·34	3·91	3·01

Variations in the concentration of excess sodium hydroxide and of coupling component in solution do not affect the diffusion coefficient appreciably (Table III).

TABLE III

Variation of Liquor Concentrations

(Brenthol AS at 25°C.)

EFFECT OF CONCENTRATION OF COUPLING COMPONENT (0·025 M. excess NaOH)

Concn. of soln., 10^3 M.	6·40	3·10
$10^5 c_\infty$, mole/kg. ...	5·86	3·82
$10^6 D$, cm.²/sec. ...	2·54, 2·33	2·38, 2·45
Mean	2·43	2·41

EFFECT OF CONCENTRATION OF EXCESS SODIUM HYDROXIDE

(Initial concn. of soln. $8·0 \times 10^{-3}$ M.)

Excess NaOH, M. ...	0·015	0·025	0·035
$10^6 D$, cm.²/sec. ...	2·29, 3·07	—	1·88, 2·72
Mean	2·68	2·40	2·30

The diffusion coefficient is sensitive to structural differences in the cellulose filaments. In Table IV a comparison is made between isotropic filament which had not been dried out after regeneration, filament dried out and reswollen once, and filament dried out and reswollen three times: no further change in properties was observed after further reswellings. The diffusion coefficient and the degree of swelling decrease on drying out and reswelling, but a stable structure is ultimately attained. Orientation of the filament by stretching reduces the diffusion coefficient; filament which had not been dried after regeneration was stretched mechanically under water and allowed to dry under tension, then reswollen three times. The extension ratio is the final dry length/dry length of an equal original wet length of unstretched filament.

TABLE IV

Effect of Modification of Cellulose

(0·005 M. Brenthol AS + 0·025 N. excess NaOH at 25°C.)

Preparation of Sample	Extension Ratio	Degree of Swelling	$10^6 D$ (cm.²/sec.)	
Never dried ...	1·0*	4·4	23·4	
Dried, reswollen once	1·0	2·1	5·03	
Dried, reswollen 3 times	1·0	1·95	2·76	
Stretched	1·39	—	2·25 2·21	2·23
Stretched	2·37	—	2·81 1·94	2·38
Stretched	2·48	—	2·11 1·97	2·03
Stretched	2·87	—	1·70 1·86	1·78

* Isotropic

TABLE V

Diffusion Coefficients and Energies of Activation

(0·025 N. excess NaOH; isotropic reswollen filament; $c_\infty \simeq 5 \times 10^{-5}$ moles/kg.)

Brenthol	Filament Batch	$10^6 D$ (cm.²/sec.) 25°C.	50°C.	Activation Energy (kcal./mole)
AS	A	2·42	13·8	13·2
OT	A	2·64	10·4	10·5
OT	B	4·50	11·4	8·3
FR	A	1·44	8·51	13·5
PA	A	1·16	6·87	14·0
FO	B	1·80	6·95	10·3
MN	B	2·27	7·72	9·3
AN	B	1·45	5·06	9·5

DISCUSSION

The diffusion coefficients of coupling components (Table V) are greater than those of direct dyes by a factor of 100: a comparable value for a typical direct dye (Sky Blue FF) at 60°C. is $3·4 \times 10^{-10}$ cm.²/sec. The coupling components used in these experiments are very closely related chemically, and in all cases the amide group is the only one likely to be involved in hydrogen bonding. It should therefore be easier to correlate structure and dyeing properties of these molecules than those of direct dyes, very few of which are at all closely related. However, it is evident from Table V that the differences in dyeing properties are comparatively small. The precision of individual values of the diffusion coefficient is not better than 10%: much significance cannot be attached to differences in the energy of activation for diffusion of less than 2 kcal./mole. The magnitude of the diffusion coefficient depends not only on the diffusing molecule but also on the nature of the medium through which the diffusion takes place, as is shown in Table IV. There appears to be a significant difference between the two batches of isotropic filament prepared at different times and used in Table V. The diffusion coefficient is a sensitive parameter which may be used to characterise different cellulose preparations. The present results serve to establish the order of magnitude of the diffusion coefficients of azoic coupling components in cellulose, the apparent energy of activation for diffusion, and the effect of orientation of the filament and of liquor variables on the rate of diffusion, though they are insufficiently precise to allow the effect of structural variations in the coupling components to be discerned with certainty.

* * *

We are indebted to Imperial Chemical Industries Ltd. (Dyestuffs Division) for the samples of Brenthols used.

Mr. M. J. Blears assisted with the experimental work described here. The apparatus was designed by Dr. W. Lonie. The cellulose model filament was prepared by Mr. R. Holloway.

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A Method of Isolation of a Cuticle Sheath from Wool Fibres

R. L. ELLIOTT and J. B. ROBERTS

Previous work on the wool fibre has resulted in the isolation of two membranous components, viz. the epicuticle and the cortical cell membranes, and the postulation of the presence of a third membrane, viz. the subcuticle membrane, or endocuticle. The present work describes the isolation from the wool fibre of a cuticular sheath, which has been shown to consist of epicuticle and endocuticle. The method described employs oxidation with potassium dichromate followed by extraction with ammonia. The material resistant to this treatment was examined in the optical microscope, and its morphological significance was confirmed by electron-microscopic studies of gold-shadowed residues. Electron microscopy also revealed a fibrillar and microfibrillar structure of the endocuticle, thus explaining the grooved appearance of the surface of damaged wool fibres (e.g. after wet chlorination). The sulphur content of the dichromate- ammonia-resistant endocuticle (comprising at most 2% of the original wool) is shown to be appreciably higher than that of the complete wool fibre. This sulphur is shown to be present in the form of cystine, but the resistance of the cystine in the cuticular layer to dichromate oxidation is unexplained.

Introduction

In 1885 McMurtrie [1] suggested the existence of a continuous layer below the cuticle of the wool fibre, in order to account for the tubes of cuticle material which he had obtained by treatment of wool with caustic soda. Much later, Reumuth [2] made a similar suggestion, based on observations of cross-sections of damaged wool fibres. In view of the importance of such a membrane for the dyeing properties of wool, a number of attempts to isolate this constituent have been reported [3–5]. Thus Lehmann [3] and Elöd and Zahn [4] claimed to have isolated such a membrane, and later Alexander and Earland made a similar claim [5]. Mercer and the Swedish workers [6a, 6b, 7] subjected these and other preparations to electron-microscopic examination, and concluded that the Lehmann tubes and the material isolated by Elöd and Zahn were in fact composed of epicuticle thickened by adhering protein. The material isolated by Alexander and Earland [5] was shown to be morphologically heterogeneous, containing material from the cortex in addition to cuticle material. As a result of their observations Mercer *et al.* [6a, 6b] suggested that the complex results obtained by various workers could be explained on the basis of a cuticle structure consisting of three components, viz. the epicuticle, the exocuticle, and the endocuticle (Fig. 1). Studies of the material first isolated by Alexander and Earland [5] have been made by Mercer [8], Manogue, Elliott, and Moss [9–11], and Fraser and Rogers [12].

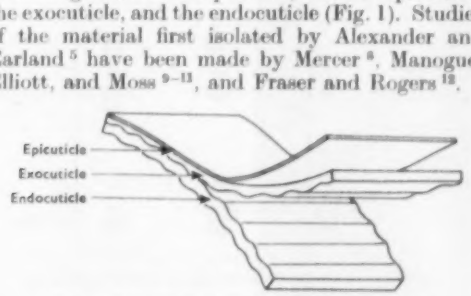

FIG. 1—Diagram of the Cuticle of the Wool Fibre [6a]

Optical-microscope studies by Mercer [8] indicated that the major constituent of these residues was a network of cortical membranes, originally surrounding each cortical cell. Electron-microscopic observations of the same material by Manogue, Elliott, and Moss [11] confirmed Mercer's [8] results. Other work by Manogue and Elliott [9] indicated that the residues consisted of this network of membranes, containing some resistant fibrous material, the whole being surrounded by a fibrous cuticle sheath, i.e. the endocuticle.

In a recent optical-microscope study of Alexander and Earland's material, Fraser and Rogers [12] have suggested a more complex structure for the cuticle than that proposed by Mercer *et al.* [6a, 6b], invoking the presence of several resistant components in the cuticle.

Also Lagermalm [13] has suggested the presence of two layers in the exocuticle, on the basis of his electron-microscope studies of the epicuticle.

The present work arises from an investigation of the action of potassium dichromate on the wool fibre. The investigation was carried out because of the industrial use of potassium dichromate in the dyeing of wool, and the known effects of such dichromate dyeings on handle and other physical properties of wool.

Experimental

I. MICROSCOPICAL INVESTIGATION

(a) *Optical Microscopy*

In order to determine whether or not the fibre could be rendered completely soluble in ammonium hydroxide by oxidation with dichromate, degreased 64s merino wool fibres (2 g.) were treated with 100 ml. of a 5% solution of potassium dichromate in N. sulphuric acid at 17°C. for 18 hr. After oxidation the fibres were washed in distilled water and extracted with 2N. ammonium hydroxide. The residues were then stained with Methylene Blue, and examined under the optical microscope (Fig. 2). The material was completely non-birefringent under polarised light.

In order to determine the original site in the wool fibre of the material shown in Fig. 2, degreased 64s merino wool fibres (approx. 1 g.) were immersed in a 5% dichromate solution in N. sulphuric acid, and samples of the fibres were withdrawn at intervals. The samples were extracted with dilute ammonium hydroxide, and examined under the optical microscope. It became obvious from this investigation that the resistant material was derived from the cuticle of the fibres. The oxidation process was therefore prolonged to 18 hr., and the oxidised wool was rinsed and extracted with 2N. ammonium hydroxide with the minimum of agitation. Fig. 3 is an optical micrograph of the residues obtained. From the scale markings on the residues it is obvious that the resistant material is derived from the cuticle.

Attempts were made to study cross-sections of the material by cutting sections of the oxidised wool and extracting these with ammonium hydroxide[cf. 11]. However, on attempted extraction, the residues disintegrated, making valid observations impossible.

(b) *Electron Microscopy*

Electron microscopes used in this investigation were Metropolitan Vickers models EM 2 and EM 3A.

A small sample of the dichromate-resistant residues was purified by repeated centrifuging and resuspension in distilled water. A small drop of the final suspension was dried down on to a filmed grid, and the specimen was then examined in the electron microscope. It was seen that the residues contained a fairly thick scale-bearing material (Fig. 4) together with a quantity of epicuticle (Fig. 5). The scale-bearing material appears to be fibrous, and is considered identical with endocuticle.

Further specimens were prepared and gold-shadowed at an angle of 30°.

Examination of these specimens indicates a fibrous structure of the endocuticle (Fig. 6 and 7), the fibrils being approx. 0·15 μ. in diameter. It would also appear (Fig. 7) that the 0·15-μ. fibrils are composed of small microfibrils, of diameter ca. 0·05 μ. (500 A.).

During the microscopical investigation, no material other than epicuticle and endocuticle was observed.

2. CHEMICAL INVESTIGATION

In order to obtain sufficient material for analysis, 150 g. of degreased 64s merino fibres were treated at 18°C. for 20 hr. with 5 litres of a 5% solution of potassium dichromate in N. sulphuric acid. The oxidised wool was filtered off, and then extracted repeatedly with 2N. ammonium hydroxide. The insoluble material was filtered off, washed with distilled water, dilute acetic acid, and finally distilled water, and dried in an oven at 100°C.

The residues were obtained as a pale grey powder weighing 1·5 g. Allowing for loss of material during isolation, this means that the residues comprised at most 2% of the original wool.

(a) *Determination of Sulphur Content*

The sulphur content of 0·1–0·2 g. samples of the cuticle sheath was determined by sodium peroxide–potassium hydroxide fusion. Before precipitation of the SO_4^{2-} so formed as barium sulphate, the liquor was boiled with 100 ml. of ethyl alcohol in order to reduce any chromate present (which would interfere with barium sulphate precipitation) to chromium salts (which do not interfere). Six determinations were made of the sulphur content of the original wool, and of that of the cuticle sheath. The means of the results obtained were as follows—

	Mean Sulphur Content (%)
Original wool	3·9
Cuticle sheath ...	6·30

(b) *Paper Chromatography*

A sample of the cuticle sheath was hydrolysed with boiling 5N. hydrochloric acid for 24 hr. An equal weight (ca. 0·5 g.) of degreased 64s merino fibres was hydrolysed in the same way. The hydrolysates were separately evaporated to dryness *in vacuo*, and each was taken up in 5 ml. of 10% aqueous *iso*propanol. Aliquots (2·5 μl.) of these solutions were taken and run on simultaneous one-dimensional chromatograms together with authentic samples of known amino acids. The solvent used was a mixture of butanol (4 parts), water (5), and acetic acid (1). The upper layer of the two-phase system was used for developing the chromatogram, and the lower layer provided the atmosphere in the chamber.

On developing the chromatograms with ninhydrin, it was found that the cystine–cysteic acid spot in the cuticle sheath hydrolysate was much larger than the corresponding spot in the 64s merino hydrolysate (Fig. 8). This is to be expected

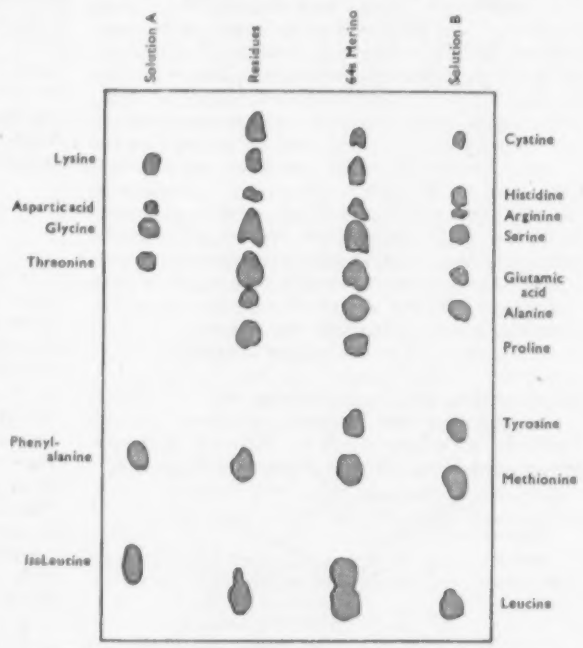

FIG. 8—Chromatograms of Cuticle Sheath and 64s Merino Hydrolysate

from the high sulphur content of the cuticle sheath. Also, it can be seen that tyrosine is completely absent from the cuticle sheath hydrolysate, probably owing to oxidation, during the isolation process, of any tyrosine originally present.

In order to determine whether any cystine remained unoxidised in the cuticle sheath, a further one-dimensional chromatogram was run, this time using as solvent the ethyl methyl ketone–propionic acid–water mixture of Clayton and

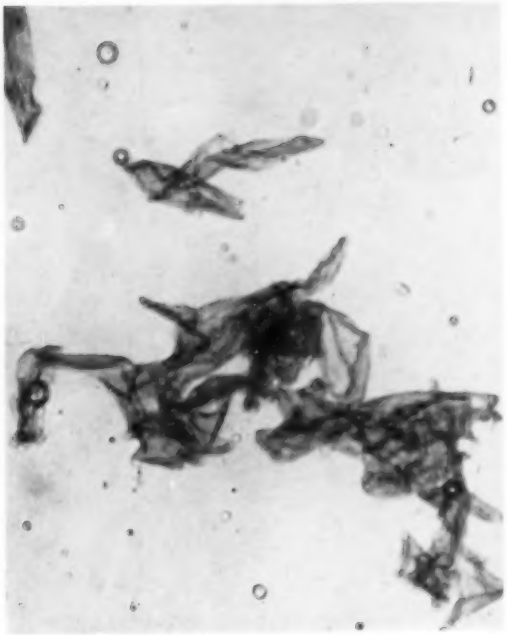

FIG. 2.—Dichromate–Ammonia-resistant Material × 600 (optical micrograph)

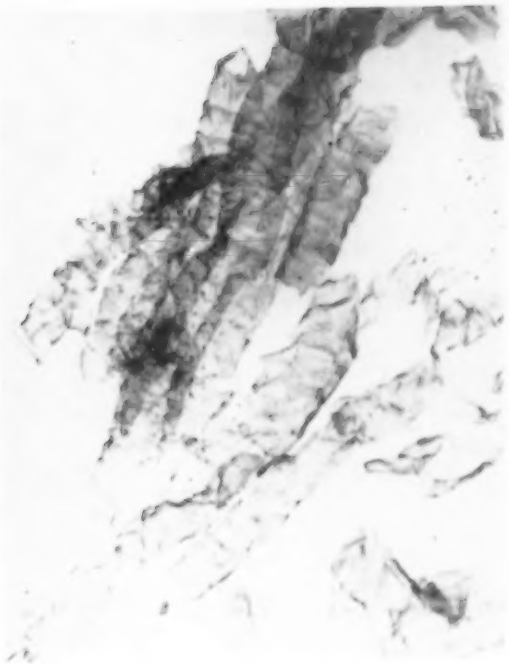

FIG. 3.—Dichromate–Ammonia-resistant Material × 250

FIG. 4.—Endocuticle in Dichromate–Ammonia-resistant Material × 2,500 (electron micrograph)

FIG. 5.—Epicuticle in Dichromate–Ammonia-resistant Material × 2,500 (electron micrograph)

FIG. 6.—Gold-shadowed Endocuticle (electron micrograph, × 10,000 approx.) (positive print)

FIG. 7.—Gold-shadowed Endocuticle (electron micrograph, × 25,000 approx.) (negative print)

Strong [14]. This solvent enables cystine to be separated from cysteic acid.

After colour development of the chromatogram, it was found that a large cystine spot was present, with only a small cysteic acid spot (Fig. 9).

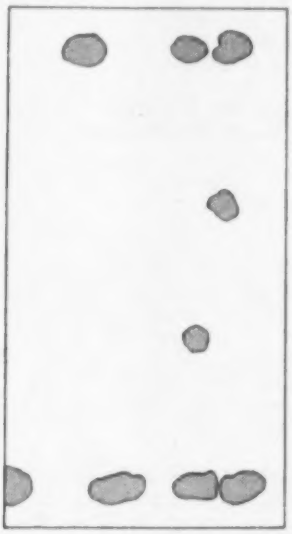

FIG. 9.—Chromatogram of Cuticle Sheath

The reason for the resistance to oxidation of the cystine in the cuticle sheath is unknown, but the presence of the cystine accounts for the insolubility of the sheath in dilute ammonium hydroxide, for the disulphide cross-links will prevent dispersion and subsequent dissolution of adjacent peptide chains.

Discussion

It has been found that oxidation of wool with acid potassium dichromate solution, followed by extraction with dilute ammonium hydroxide, leaves an insoluble residue of high sulphur content, comprising at most 2% of the original wool. Microscopical investigations of the residue have revealed that it is a cuticle sheath consisting of epicuticle and endocuticle.

Electron-microscope studies of the endocuticle have shown it to be composed of fibrils 0·15 μ. in diameter, each fibril being built up of microfibrils 0·05 μ. in diameter.

The fibril structure of the endocuticle is responsible for the grooved surface of damaged wools, and the microfibrils are responsible for the fine grooved structure which has been observed on alkali-damaged wool [15]. The fibrils and the microfibrils may also be indirectly responsible for two types of structures, one coarse and one fine, observed by Lagermalm [13] on epicuticle.

The high sulphur content of the cuticle sheath agrees with the results of Geiger [16], who isolated cuticle material by reduction, ethylation, and enzymatic digestion of the wool fibre.

* * *

The authors wish to thank the Wool Textile Research Council for financing this work, and for the award of a Scholarship to J.B.R.

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TECHNICAL COLLEGE
BRADFORD 7
YORKSHIRE

(*Received 18th September 1956*)

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[11] Manogue, Moss, and Elliott, *J.S.D.C.*, **70**, 502 (1954).
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[15] Ingamells, private communication.
[16] Geiger, *Bur. Stand. J. Res.*, **32**, 127 (1944).

CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Rapid Dyeing

H. à Brassard's letter [1] poses a very interesting problem, which involves many detailed experiments, because each dye is an individual as regards rapid dyeing. In my experience direct dyes which are good for variable viscose rayon show rapid-dyeing properties. The introduction of Rigan Sky Blue G by Ciba gave me the first lesson in rapid dyeing. We confirmed in the laboratory Ciba's claims, and our first bulk dyeing was 600 lb. of viscose rayon skeins on an 80-roller Gerber machine. To our shocked surprise every skein was ended. The keen observation of my head dyer, C. Wilkinson, gave the explanation: when the Gerber rollers were lifted out of the dye liquor for unloading, he noticed that the last drainings off the skeins were colourless, whereas the dye liquor in the dyebath was still strongly coloured. This showed that rapid dyeing had taken place as the dye liquor on the skeins had slowly drained: this took place in 3 min. or so. A simple experiment in the laboratory confirmed this. Dye a skein in a basin, then lift it out, and without wringing let it lie for 3–5 min. in another basin; wring the liquor out of the skein into a third basin and compare it with the dye liquor in the first basin: it will always be much more exhausted. The trouble was cured by dyeing to near exhaustion before raising the Gerber rollers. Normally we never dyed direct dyes to exhaustion, which was considered more favourable for covering unevenly dyeing viscose rayon.

Intelligent application of rapid dyeing enables the time of dyeing of viscose rayon staple to be considerably reduced.

C. M. Whittaker

Red Lea Hotel
Scarborough

24th January 1957

[1] J.S.D.C., **73**, 24 (Jan. 1957).

"Man-made"

With reference to Mr. Marsh's suggestion [1] of the term "manufil", I remain of the opinion that the fibres under discussion are "chemical" fibres [2], and therefore would prefer *chemifil*. And I continue to submit that a product cannot fairly be called "synthetic" unless it is the result of a synthesis of a natural product.

H. à Brassard

136 Grove Hall Court
Hall Road
London N.W. 8

29th December 1956

[1] Marsh, J. T., J.S.D.C., **72**, 577 (1956).
[2] à Brassard, H., *ibid.*, **69**, 496 (1953).

Notes

The Second Edition of the *Colour Index*

Volume 1 of the Second Edition of the *Colour Index* was published at the end of 1956, by the Society in Bradford and by the AATCC in Lowell, Massachusetts. The other three volumes will be published later, in 1957. Volumes 1 and 2 together constitute Part I, which classifies dyes according to their application groups. Volume 3 will classify dyes according to their chemical constitution, and Volume 4 will consist mainly of a general index.

This outstanding event represents the beginning of the culmination of a decade of voluntary labour on both sides of the Atlantic.

New *Colour Index* Numbers

As Volume 1 of the Second Edition of the *Colour Index* has now been published, and the other volumes are expected within the next few months, the new *C.I.* numbers will be quoted where necessary in the *Journal* beginning with the present (March 1957) issue. In general the usage numbers, e.g. C.I. Acid Red 35, will be given, as they are probably more informative; but in some cases a five-figure number referring to the chemical constitution of a dye, e.g. C.I. 68205, will be quoted. It will be seen that there will be no danger of confusion with numbers referring to the First Edition of the *Colour Index*.

Fred Smith, F.S.D.C.
Honorary Member of the Society

At the meeting of Council held on 6th February 1957 it was unanimously resolved, on the recommendation of the President, that Honorary Membership, the highest honour that the Society can bestow, be accorded to Mr. Fred Smith.

Mr. Smith began his many years of service to the Society in 1915 as a member of the West Riding Section Committee, and was President during 1954–1956.

Cyril Oldtree Clark, F.T.I., F.S.D.C.
Gold Medal of the Society

On the recommendation of the Society's Medals Committee, Council has unanimously resolved that the Society's gold medal be awarded to Mr. C. O. Clark in recognition of his outstanding services to the Society.

Mr. Clark began his long period of service to the Society as Honorary Secretary of the Bradford Junior Branch. He has served for many years on the Publications Committee and as Editor of Abstracts of Patents for the *Journal*. He is Chairman of the Examinations Board, Joint Co-ordinator of the *Colour Index* Editorial Panel, and a member of Council.

Charles Lawrence Bird
M.Sc., F.R.I.C., F.S.D.C.
The Worshipful Company of Dyers Research Medal 1955-1956

On the recommendation of the Council of the Society, the Worshipful Company of Dyers has awarded its Research Medal for 1955–1956 to C. L. Bird, as senior author of the series of papers on *The Dyeing of Acetate Rayon with Disperse Dyes*.

Meetings of Council and Committees
February

Council— 6th
Finance* and General Purposes— 6th
Perkin Main— 13th
Publications— 19th
Textbooks and Monographs Subcommittee— 19th
Colour Index Editorial Panel— 21st

Deaths

We regret to report the loss by death of Mr. A. Chadwick and E. N. Walters.

Perkin Centenary Awards 1957

Applications for these awards (see J.S.D.C., **73**, 57 (Feb. 1957)) should be submitted by **1st May 1957.** Copies of the regulations and forms of application may be obtained from the Secretary, the Perkin Centenary Trust, c/o the Chemical Society, Burlington House, London W.1.

Donation of Journals by the Chemical Society

The Chemical Society has been kind enough to give our Society fairly long runs of past issues of two foreign periodicals of special interest to us.

The first is "Matières Colorantes" (*Revue Générale des Matières Colorantes, de la Teinture, de l'Impression et des Apprêts*), volumes **32–43**

(1919–1939). The early bound volumes contain also issues of *Le Teinturier Pratique*, *Revue Générale de l'Industrie Textile*, and *Revue Mensuelle du Blanchissage, du Blanchiment et des Apprêts*. The run actually finishes with the May 1940 issue (volume **44**, No. 520), but the issues for December 1939 and January and April 1940 are missing. This journal is now better known as *Teintex*, which has reached volume **22** in 1957.

The other periodical is *Farben-Zeitung*, which has the subtitle "Fachblatt für die gesamte Farben- und Lack-Industrie sowie den einschlägigen Handel". The run is complete from 4th October 1924 (starting volume **30**) to 26th August 1939 (No. 34 of volume **44**); the issues dated 23rd and 30th December 1939 are also included.

We are most grateful to the Chemical Society, the resources of whose Library are freely available to our members, for thus adding to our collection of literature relating to colour and coloration.

Dr. Henry Phillips, O.B.E.

The Order of the British Empire has been conferred on Dr. H. Phillips, Director of Research of the British Leather Manufacturers Research Association. Dr. Phillips was awarded the Research Medal of the Worshipful Company of Dyers for 1946–1947.

New Films

Three new films have recently been added to the I.C.I. Film Library. *Point of New Departure* (16 mm., colour, sound, 35 min.) is a non-technical lecture by Mr. J. R. Whinfield on the evolution of man-made fibres. *Terylene on Parade* shows a fashion parade, and *More about Alkathene* shows some of the less well known uses of polythene. The two latter films are black and white (16 mm., 5 min.). All three films may be borrowed from the I.C.I. Film Library, Imperial Chemical House, Millbank, London S.W.1.

A non-technical sound film has been made in America to explain the physics of colour. The film has been photographed in Eastman Color and it runs for 27 min. Arrangements to borrow it may be made through Interchemical Corporation, 67 West 44th Street, New York 36, N.Y., U.S.A.

Synthetic Detergents

The Minister of Housing and Local Government has appointed a Standing Technical Committee on Synthetic Detergents, consisting of representatives of manufacturers of detergents and river and water authorities. The Committee's terms of reference are as follows—

> To keep under review the difficulties, or risks of difficulty, arising in sewage works, rivers and water supply as a result of the use of synthetic detergents; to encourage, and assist the co-ordination of, appropriate research by manufacturers of detergents and intermediate materials, and by suitable public bodies, into methods by which those difficulties or risks of difficulty, could without an undue burden on public funds be avoided or overcome; and to report progress at least once a year.

New Books and Publications

Perspectives in Organic Chemistry

Edited by Sir Alexander Todd. Pp. x + 527. New York and London: Interscience Publishers. 1956. Price, $7.50.

This book reflects a spread of the German custom of honouring the decades in the lives of the famous, for it has arisen from the desire of a group of friends, colleagues, and pupils of Sir Robert Robinson to honour his seventieth birthday in September 1956, appropriately the Perkin centenary year. It is a collection of personal and speculative essays, rather than detailed reviews, by leading workers in their own fields. Whether because of Sir Robert's own interests, or because of the importance of dyes and dyeing to organic chemistry in general, a surprisingly large proportion of the book is relevant to the field of interest of the Society.

In the first essay Linus Pauling stresses that the theory of resonance was originally based essentially on classical valency structures (in cases where more than one arrangement of bonds is required to represent the properties of the compound), and preceded its interpretation in terms of quantum mechanics. One of the most important applications of resonance theory, aromaticity, is discussed by Wilson Baker, who foresees the possibility of larger conjugated macrocycles analogous to phthalocyanine. In an interesting account (in German) of large rings, V. Prelog suggests that the amazing ease of formation of this compound depends on the geometry of the monomer. A theme running through this and several other essays, e.g. those on reaction mechanisms and stereochemistry, is the importance ascribed in modern organic chemistry to the actual spatial distribution or conformation of the atoms, even hydrogen atoms, in a molecule.

Several essays deal, as is to be expected, with natural products, which, it is interesting to learn, include a diazo compound. E. L. Hirst compares the structure of straw and wood with that of reinforced concrete, "ropes" of cellulose being embedded in an amorphous mass of lignin and polysaccharide material. The account by Sir Alexander Todd of nucleic acids—linear polyesters—is of absorbing interest, since they are the basis of us: a double coaxial helix structure for deoxyribonucleic acid (with the protein forming a third coaxial chain) may begin to explain hereditary transmission from cell to cell, the mutation of genes, and the control of protein synthesis in the living cell, owing to the necessity for conformation of groups forming hydrogen bonds between the chains. An interesting modern development is the working out of syntheses,

e.g. of alkaloids, "under physiological conditions", viz. in near neutral aqueous media at ordinary temperatures. Perhaps there is a lesson for the dyemaker here—several hydroxyanthraquinones are mould metabolites, whose oxidation and reduction may play a rôle in antibiosis; and phenoxazones, made for sixty years in industry, have recently been isolated from the visual pigments of insects.

The essay by James Walker on chemotherapy stresses the experimental difficulties and also the fact that many advances have depended on the use of dyes. Thus the selective staining of tissues made it natural that Ehrlich should turn to dyes for the selective killing of organisms, e.g. trypanosomes. The development of the sulphonamide drugs can be traced directly to the belief that the sulphamoyl $SO_2{\cdot}NH_2$ group improves the fastness of azo dyes on wool.

The above examples can give only a faint idea of the great interest of this collection of essays, dedicated to a distinguished Honorary Member of the Society. The publishers are to be congratulated on their enterprise, but it makes the reviewer wince a little that this, of all books, should have been published in New York.

C.J.W.H.

Fibres, Plastics, and Rubbers
A Handbook of Common Polymers

By W. J. Roff. Pp. xvi + 400. London: Butterworths Scientific Publications. 1956. Price, 63s. 0d.

The field of polymer science is now so vast that any book which assists the practical chemist in correlating the factual data is always welcome. Mr. Roff can be congratulated on the thorough way in which he has performed the arduous task of making such data, on fibres, plastics, and rubbers, easily accessible.

The book is divided into two parts, in the first of which the manufacture, properties, etc. of each type of polymer are considered in turn. Though the book is termed "A Handbook of Common Polymers", the author has introduced nearly every polymer which has the slightest possibility of application; thus even such rarities as polyphosphonitrilic chloride receive some mention. The book is not confined to the synthetic polymers but deals also with natural materials such as cellulose, protein fibres, and rubber. Each important polymer type is dealt with in a separate table, which gives its structure, manufacture, chemistry, physical properties, uses, and history.

By clear subheading and concise statement an immense amount of valuable information has been compressed into very small space. In some cases the reader may feel that this process is carried too far: e.g., whilst Acrilan and Orlon are given as trade names for polyacrylonitrile fibres, no mention is made of their different dyeing properties, except a general statement that acrylonitrile can be copolymerised with various materials which improve its affinity for dyes.

The second part of the book consists of chapters which define and give methods of measurement of the properties mentioned under the separate polymers. Thus there are short chapters on molecular weight determination, methods of identification, solvents, plasticisers, dielectric constant, etc. The chapters of particular interest to the readers of this *Journal* are those relating to fibre properties, e.g. moisture regain, wet and dry strength, elasticity, extension at break, and refractive index. Each chapter concludes with a list of values for the polymers mentioned in Part I. This is useful for rapid comparisons and facilitates reference to a particular topic.

References are given throughout the text, but the emphasis is on books rather than original papers. Although the books are well chosen, occasionally original references might be more desirable; e.g. in a table of amino-acid contents of proteins, no references are made to the sources of the values.

Misprints and factual errors are nearly non-existent, and the book will be welcome to all chemists who have the slightest connection with any aspect of high-polymer science.

R. S. Asquith

An Introduction to Modern Organic Analysis

By Sidney Siggia and Hans J. Stolten. Pp. vii + 250. New York and London: Interscience Publishers. 1956. Price, $4.50.

This book does not set out to be an analytical laboratory textbook, but to enumerate and describe a number of physical methods which are of use to the organic analyst. It is not intended that the reader shall be told how to carry out a particular analysis but rather that he be informed about the methods available to him.

The majority of the chapters are each devoted to a single class of procedures, such as methods of separation, freezing-point determination, absorption spectroscopy, and X-ray diffraction. So-called specialised approaches, such as microscopy, mass spectrometry, and Raman spectroscopy, are dealt with briefly in one chapter. A very useful final chapter is devoted to the interrelations between different analytical approaches. It takes the form of a number of actual analyses in which a variety of techniques were used to obtain the final result.

What the authors have set out to do is unusual and laudable. Unfortunately, the result does not quite come up to expectations, and the reader is left feeling rather unsatisfied and frustrated. But perhaps this is simply because the authors are attempting a new kind of presentation, with which the average chemist is unfamiliar. Even so, a closer attention to details of composition would have helped. Thus, rotational, vibrational, and electronic spectra are all discussed—albeit briefly—in one long paragraph. Some division here would make for easier reading.

However, this is not to detract from the credit due to the authors for attempting to fill a gap in the analytical literature. Their declared objective is ". . . to describe these available tools, their fundamental principles and techniques, their correlation, their application, and the interpretation from the viewpoint of the organic

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ALGINATES

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I.C.I. recommend sodium alginate as the thickening agent for printing with their new Procion dyestuffs and for preventing migration of the dyestuffs during intermediate drying when dyeing by the "wet on dry" process.

MANUTEX R.S.

a readily soluble high viscosity sodium alginate, is the A.I.L. product manufactured especially for use by the textile industry.

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For Scouring, Cleansing and Penetrating all classes of textiles. Unaffected by Acids, and Alkalis, and thoroughly stable in hard water

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Unequalled in value as a mordant for basic colours on all types of materials. Only one hour required to mordant your cotton — no fixing bath required

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Prevents cotton colours from staining wool. Essential for best results with two-colour effects

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A powerful aid for grease and tar removal by spotting and in detergent baths

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PENTROSANS
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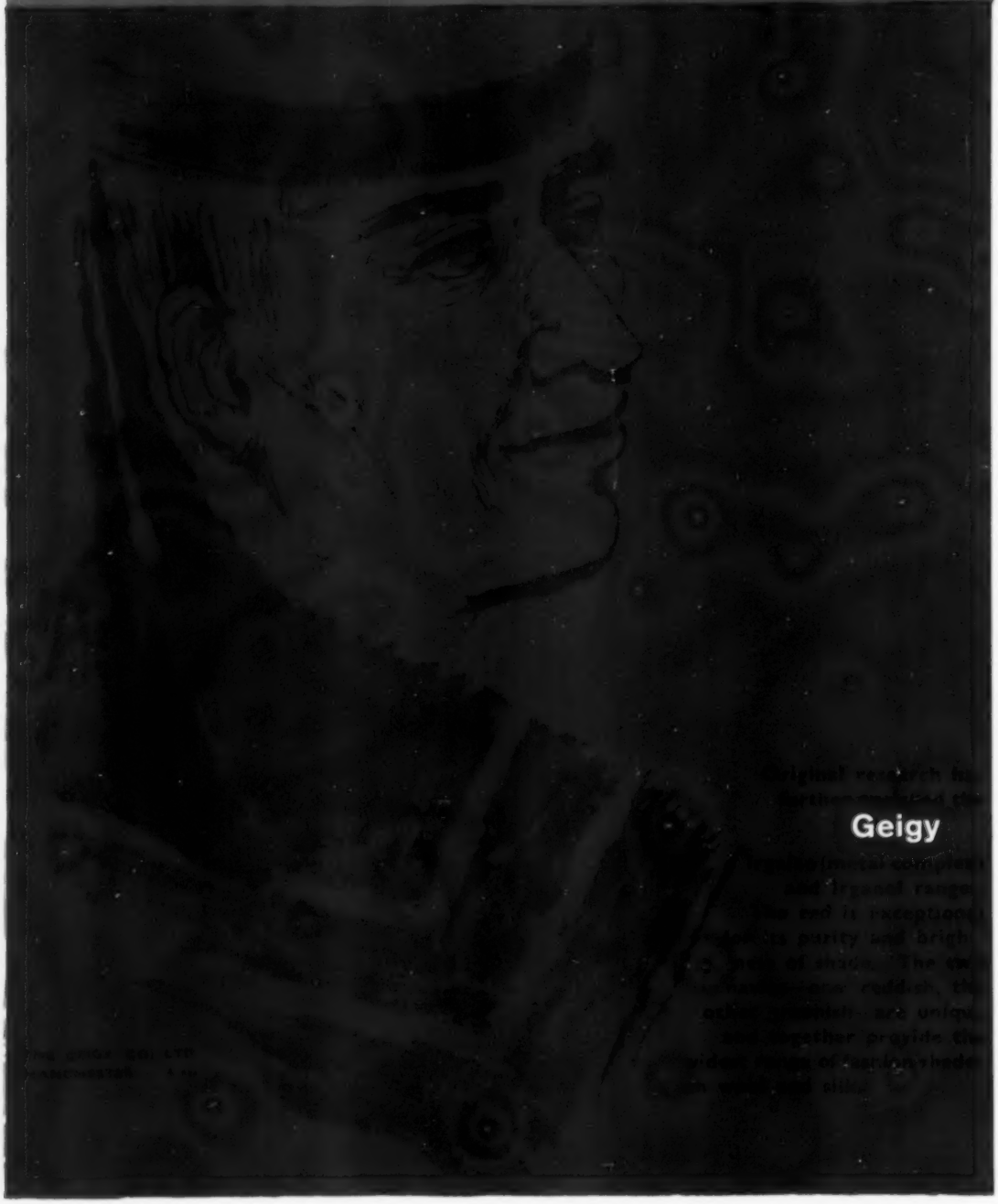
face xiv

3 Winners

Irgalan Red 2BL

Irgalan Navy Blue 5RL

Irganol Navy Blue 2RLS



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SOLVITOSE C5

a new printing thickener

gives you 3 good trumps in textile printing

Readily soluble in cold water
Thickenings are ready in a few minutes. Simply pour this unique thickener into cold water. No boiling, cooling or filtering required, meaning more uniform thickenings and a saving in time and money.
High colour yield
Solvitose C 5 gives the highest colour yield of starch thickeners and has definite advantages over combinations of starch and British Gum, starch and tragacanth etc for the printing of vat colours, rapidogens, indigosols, direct colours and discharge work.
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Thickenings made with Solvitose C 5 are completely removed in open-width soaping. This is of considerable value for fabrics which are given a subsequent resin finish.

No other thickening agent combines all these properties

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Agrias hewitsonius is a rare species of this beautiful family of butterflies found in the Amazon forests. It flies swiftly and timidly at noon on very hot days

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For all types of Machine, Piece, Hank and Loose Stock Dyeing. Also for the Rubber Proofing Trade, for which a wider range is now available

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SULPHOL Colours are suitable for dyeing cloths for subsequent P.V.C plastic coating, because they are insoluble in plasticisers and therefore do not migrate

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SULPHOL Colours may be applied from a Caustic Soda and Sodium Hydrosulphite bath. Details of application on request

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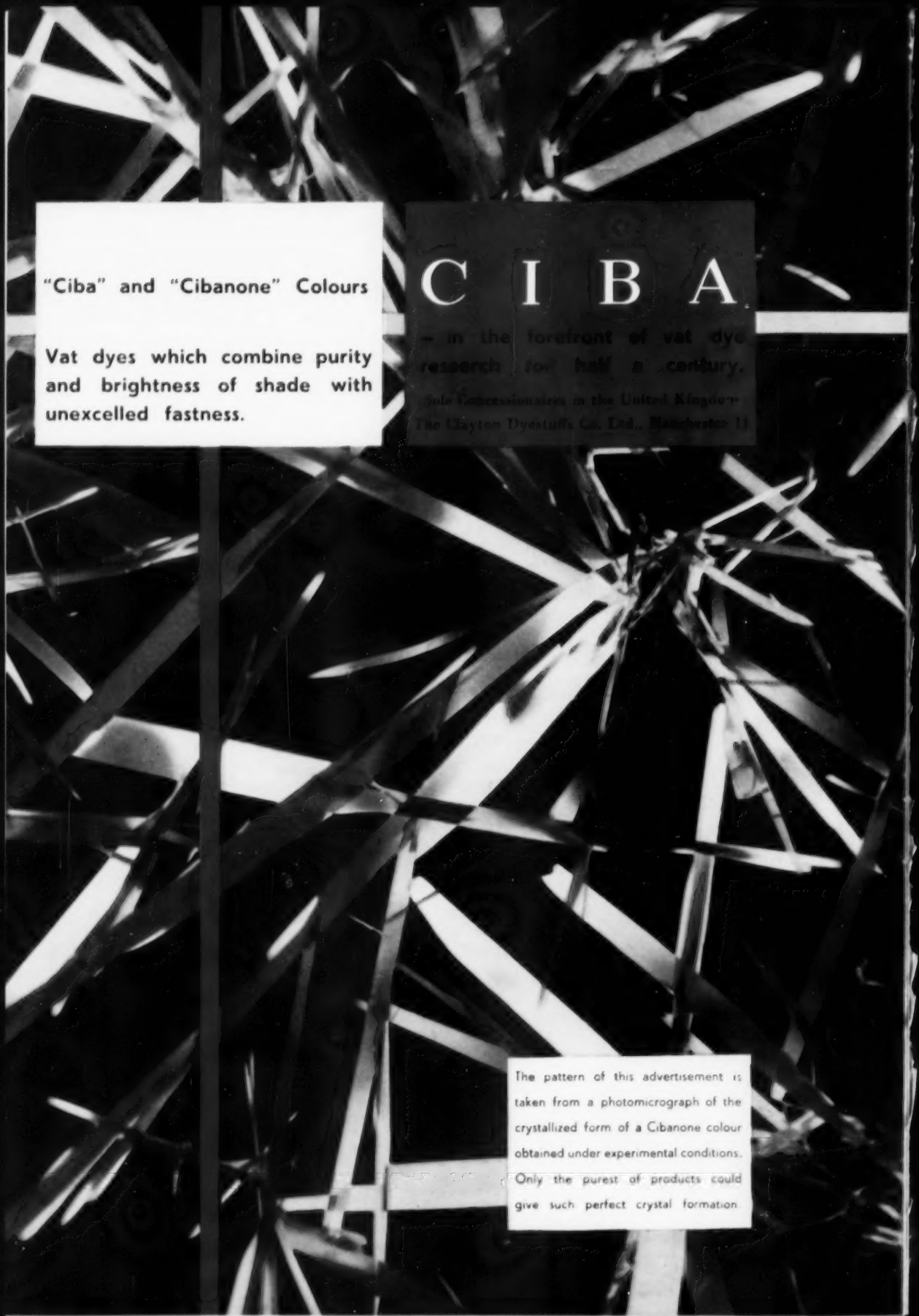
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Vat dyes which combine purity and brightness of shade with unexcelled fastness.
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Sole Concessionaires in the United Kingdom:
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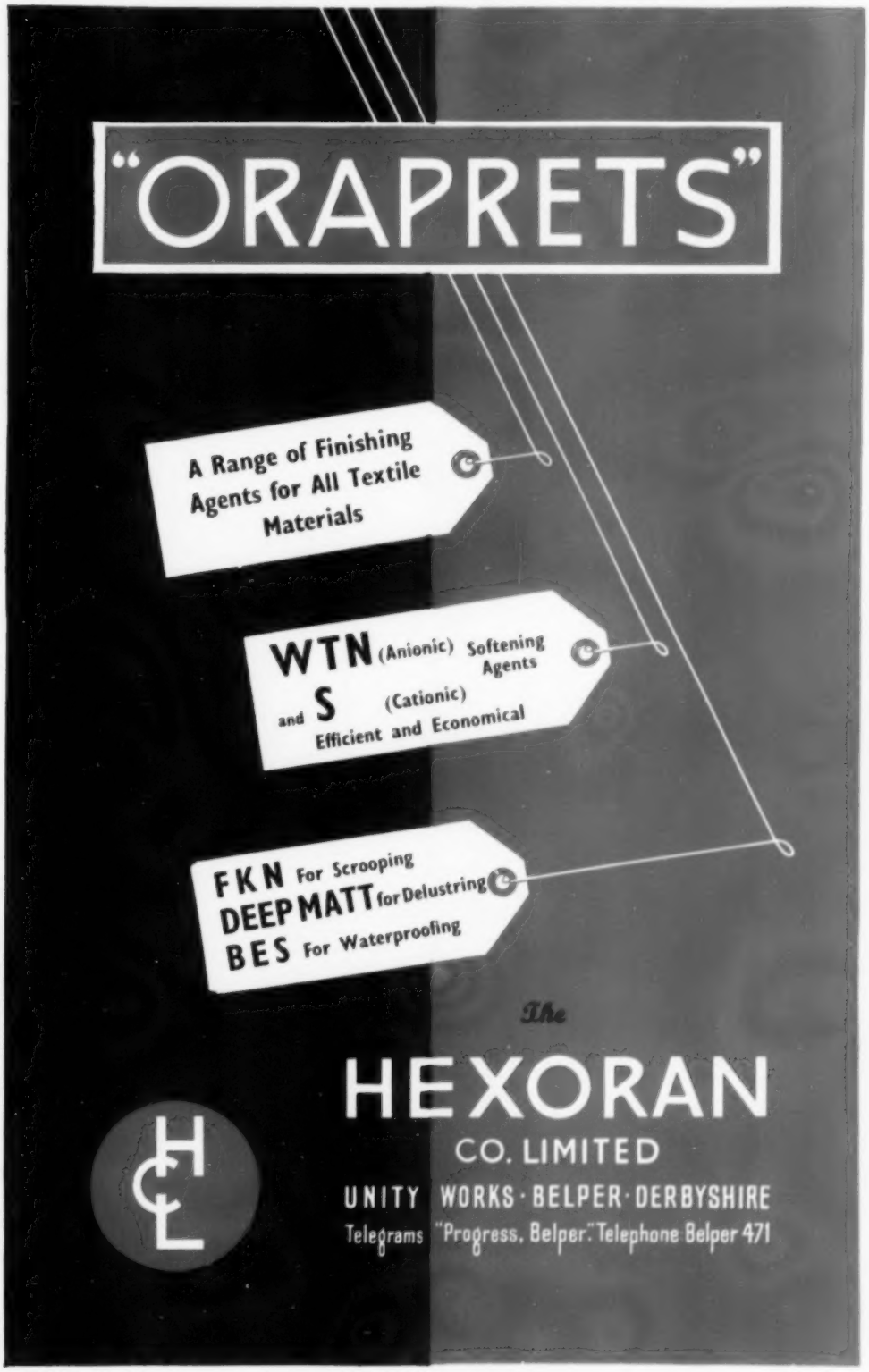
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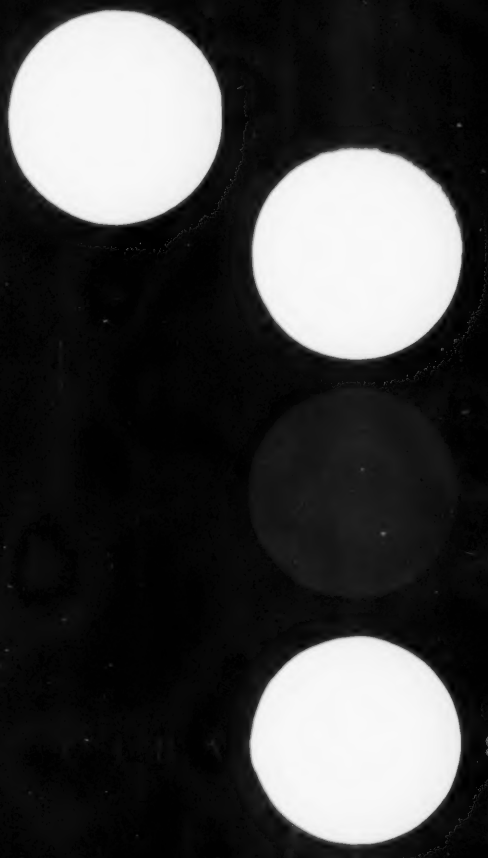
Chlorantine Fast Blue B5GL

Withstand treatment with synthetic resin finishes
Cover viscose rayon of varying affinity
Reserve acetate rayon



Special features:

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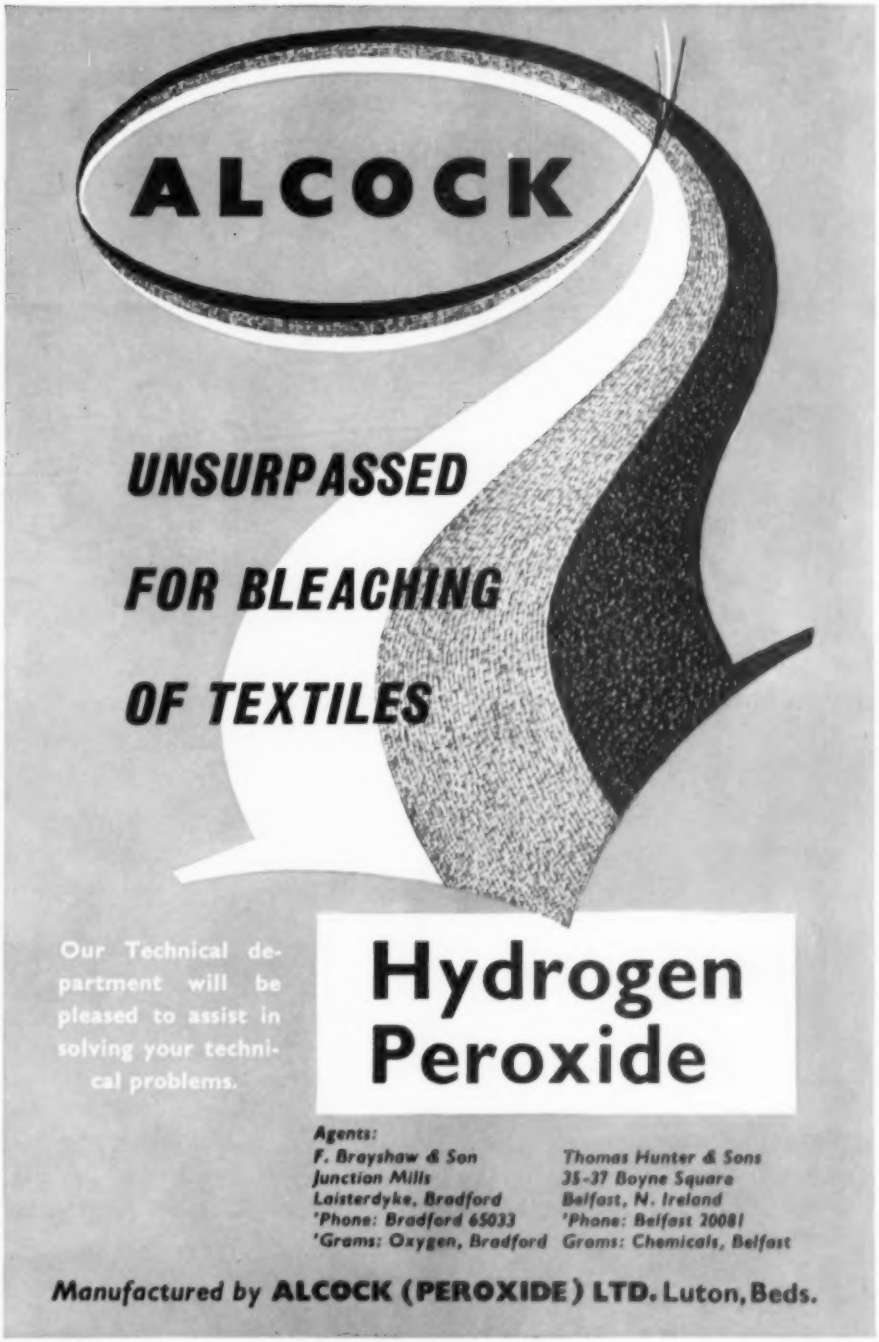
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Inns and the Textile Trade

No. 8 *'The Fleece', Kendal, Westmorland*

By permission of William Younger & Co. Ltd.

When Shakespeare was dreaming up fat Falstaff's invective at the 'misbegotten knaves in Kendal green', The Fleece at Kendal was already well-established.

The inn is at least three centuries old, though no one seems to know its exact date. We do know, however, that the trade which gave the house its name—the sheep, the fleece, the yarn that was woven into the hardwearing cloth known as Kendal green—dates back to 1331 when Edward III gave weaver John Kemp licence to work in the town.

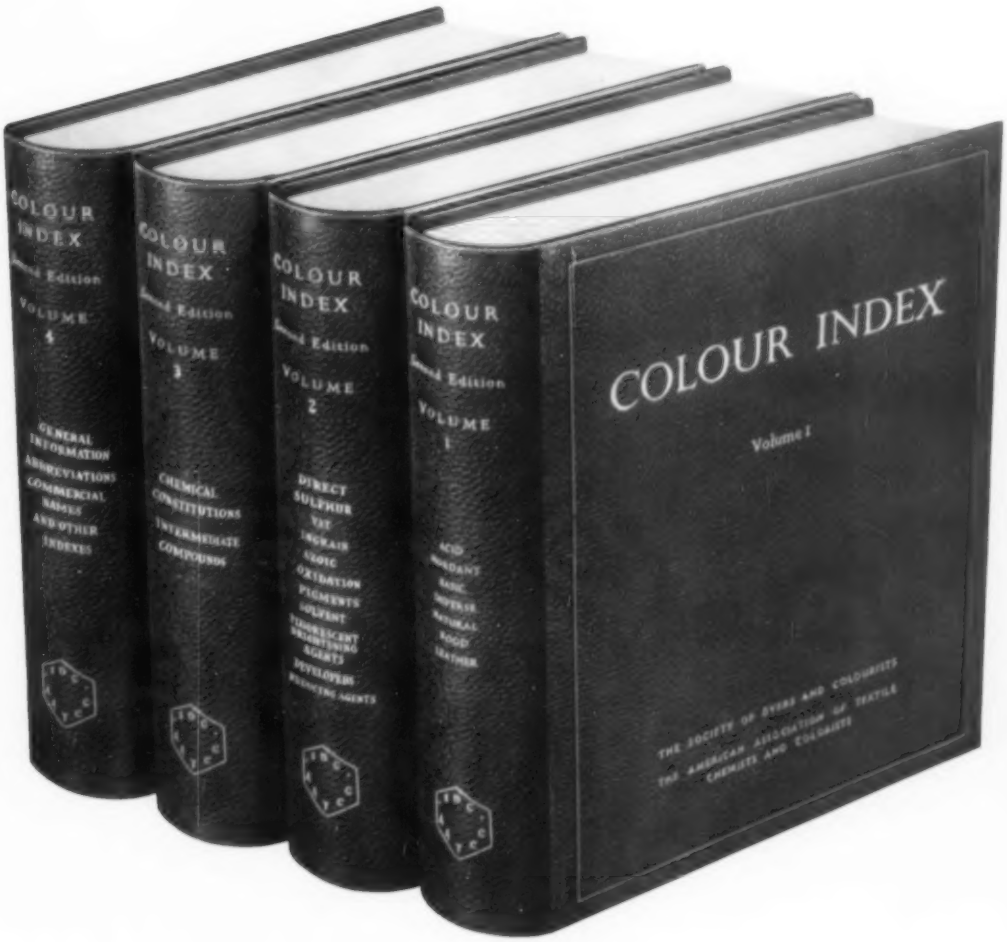
The trade flourished. To the Kendal townsfolk it became all-important. So much so that blazoned with the town's coat-of-arms is the motto '*Panus mihi panis*'—wool is my bread.

The motto could apply to all of us. Wool is still one of our nation's greatest sources of revenue. The trade, though it preserves many of its ancient traditions, is constantly being studied to meet new demands and processes are carefully modernised. Some of the chemicals necessary for this important and profitable work are supplied by Brotherton.

Brotherton

One of the world's largest manufacturers of hydrosulphites, liquid sulphur dioxide and hexamine.
Makers of a range of Metachrome dyes for dyeing wool in all its forms.
Brotherton & Co. Ltd., P.O. Box No. 6, Leeds, 1. Telephone: Leeds 2-9321. Telegrams: 'Brotherton, Leeds'.

THESE FOUR VOLUMES OF THE NEW COLOUR INDEX WILL BE AVAILABLE IN 1957



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VOLUMES 3 & 4 WILL BE AVAILABLE LATER THIS YEAR

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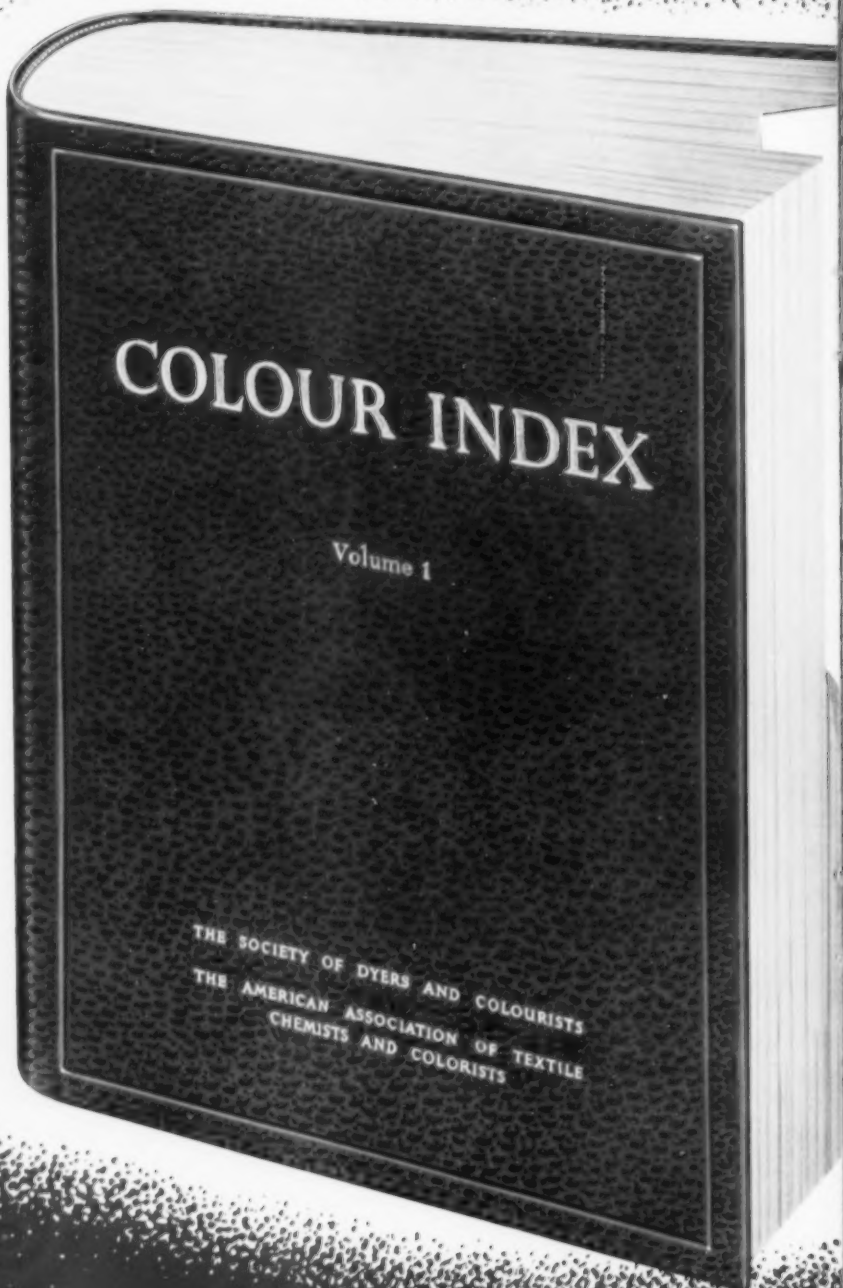

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this wonderful work...

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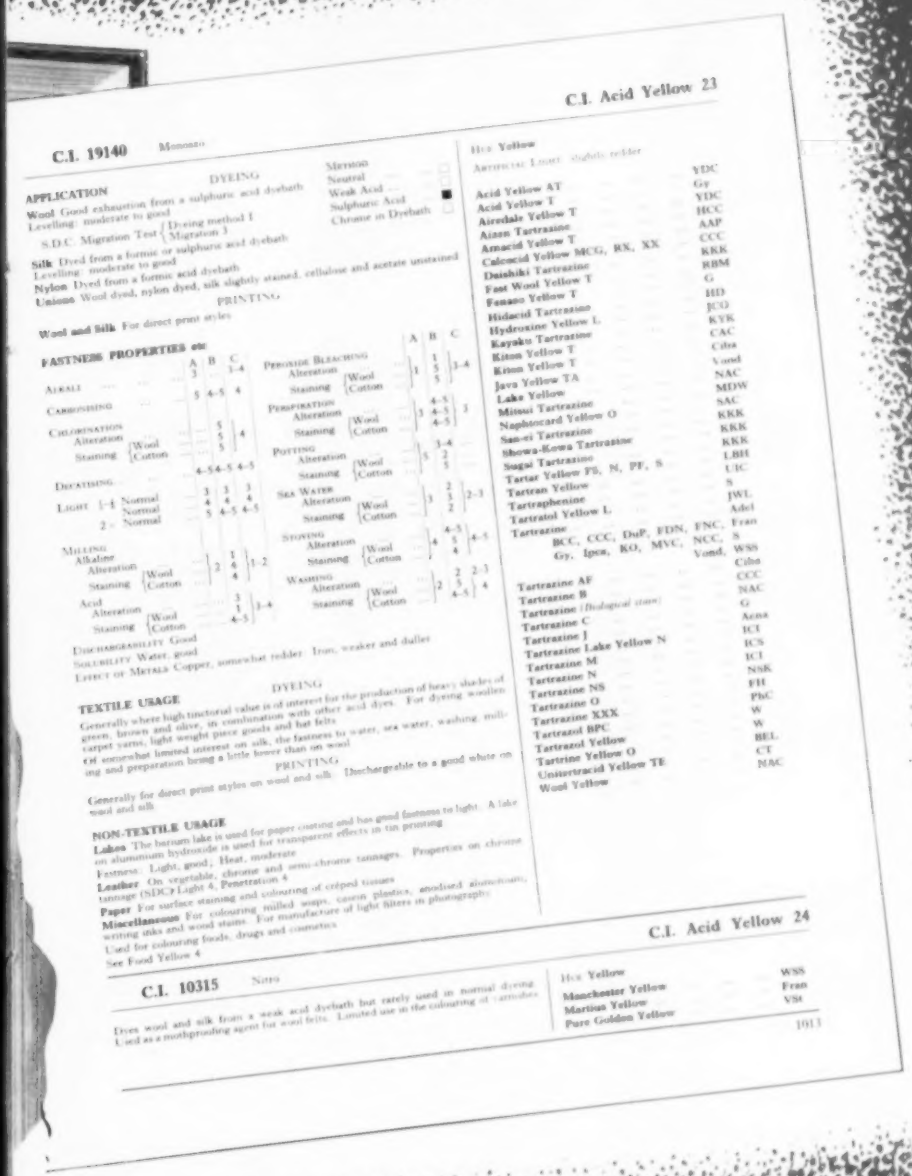

C.I. Acid Yellow 23

C.I. 19140 Monazo

APPLICATION — DYEING

Wool Good exhaustion from a sulphuric acid dyebath
Levelling: moderate to good
S.D.C. Migration Test { Dyeing method 1 / Migration 3 }

Silk Dyed from a formic or sulphuric acid dyebath
Levelling: moderate to good
Nylon Dyed from a formic acid dyebath
Unions Wool dyed, nylon dyed, silk slightly stained, cellulose and acetate unstained

PRINTING

Wool and Silk For direct print styles

FASTNESS PROPERTIES on:

	A	B	C
Alkali	3		3-4
Carbonising	5	4-5	4
Chlorination Alteration	5		
Chlorination Staining (Wool)	5		4
Chlorination Staining (Cotton)	5		
Decatising	4-5	4-5	4-5
Light 1-4 Normal	3	3	3
Light 2 - Normal	4	4	4
Light 2 - Normal	5	4-5	4-5
Milling Alkaline Alteration	2	3-4	3-4
Milling Alkaline Staining (Wool)			
Milling Alkaline Staining (Cotton)		4	
Milling Acid Alteration	3		3-4
Milling Acid Staining (Wool)	4		
Milling Acid Staining (Cotton)	4-5		

	A	B	C
Peroxide Bleaching Alteration	1	5	3-4
Peroxide Bleaching Staining (Wool)	5		
Perspiration Alteration	3	4-5	3
Perspiration Staining (Wool)		4-5	
Perspiration Staining (Cotton)		4-5	
Potting Alteration	5	3-4	
Potting Staining (Wool)		2	
Potting Staining (Cotton)		3	
Sea Water Alteration	3	2	2-3
Sea Water Staining (Wool)		3	
Sea Water Staining (Cotton)		2	
Stoving Alteration	4	4-5	4-5
Stoving Staining (Wool)		5	
Stoving Staining (Cotton)		4	
Washing Alteration	2	2	2-3
Washing Staining (Wool)		5	4
Washing Staining (Cotton)		4-5	

Dischargability Good
Solubility Water, good
Effect of Metals Copper, somewhat redder Iron, weaker and duller

TEXTILE USAGE — DYEING

Generally where high tinctorial value is of interest for the production of heavy shades of green, brown and olive, in combination with other acid dyes. For dyeing woollen carpet yarns; light weight piece goods and hat felts
Of somewhat limited interest on silk, the fastness to water, sea water, washing, milling and preparation being a little lower than on wool

PRINTING

Generally for direct print styles on wool and silk Dischargeable to a good white on wool and silk

NON-TEXTILE USAGE

Lakes The barium lake is used for paper coating and has good fastness to light. A lake on aluminium hydroxide is used for transparent effects in tin printing
Fastness Light, good; Heat, moderate
Leather On vegetable, chrome and semi-chrome tannages. Properties on chrome tannage (SDC) Light 4, Penetration 4
Paper For surface staining and colouring of crêped tissues
Miscellaneous For colouring milled soaps, casein plastics, anodized aluminium, writing inks and wood stains. For manufacture of light filters in photography
Used for colouring foods, drugs and cosmetics
See Food Yellow 4

Hue Yellow
Auramine Liquor, slightly redder

Acid Yellow AT	YDC
Acid Yellow T	Gy
Airedale Yellow T	YDC
Aizan Tartrazine	HCC
Amacid Yellow T	AAP
Amacid Yellow MCG, RX, XX	CCC
Calcocid Yellow MCG, RX, XX	KKK
Daishiki Tartrazine	RBM
Fast Wool Yellow T	G
Fenaco Yellow T	HD
Hidacid Tartrazine	JCD
Hydroxine Yellow L	KYK
Kayaku Tartrazine	CAC
Kiton Yellow T	Ciba
Kiton Yellow T	Verol
Java Yellow TA	NAC
Lake Yellow	MDW
Mitsui Tartrazine	SAC
Naphthocard Yellow O	KKK
San-ei Tartrazine	KKK
Showa-Kowa Tartrazine	KKK
Sogai Tartrazine	LBH
Tartar Yellow FS, N, PF, S	UIC
Tartran Yellow	S
Tartraphenine	JWL
Tartratol Yellow L	Adel
Tartrazine BCC, CCC, DuP, FDN, Gy, Ipca, KO, MVC,	FNC, Fran NCC, S Vond. WSS
Tartrazine AF	Ciba
Tartrazine B	CCC
Tartrazine (Biological stain)	NAC
Tartrazine C	G
Tartrazine J	Acna
Tartrazine Lake Yellow N	ICI
Tartrazine M	ICS
Tartrazine N	ICI
Tartrazine NS	NSK
Tartrazine O	FH
Tartrazine XXX	PhC
Tartrazol BPC	W
Tartrazol Yellow	W
Tartrine Yellow O	BEL
Unitartracid Yellow TE	CT
Wool Yellow	NAC

C.I. Acid Yellow 24

C.I. 10315 Nitro

Dyes wool and silk from a weak acid dyebath but rarely used in normal dyeing. Used as a mothproofing agent for wool felts. Limited use in the colouring of varnishes

Hue Yellow

Manchester Yellow	WSS
Martius Yellow	Fran
Pure Golden Yellow	VSI

1913

SDC AATCC

OUR INDEX

by placing your order Now!

THE NEW COLOUR INDEX

Produced by

THE SOCIETY OF DYERS AND COLOURISTS

THE AMERICAN ASSOCIATION OF TEXTILE CHEMISTS AND COLORISTS

VOLUMES 1 & 2 ARE NOW READY

The work has been completely re-designed and brought up-to-date with the co-operation of all the dye and pigment manufacturers in Great Britain, America, Western Europe, India and Japan.

PART I—Consists of two volumes, of which Volume 1 is now ready, and each contains approximately 1,000 pages. This part deals with all commercially homogeneous dyes and pigments in current use (approximately 4–5,000 dye entities and 25,000 commercial names) within groups relating to their main usage, e.g. Disperse, Acid, Azoic, Direct, Pigment, Solvent etc. The data included comprises the commercial name and manufacturer's initials, methods of application, fastness properties, established usages, reactions on the fibre and literature references relating to application. In the absence of a single accepted international system of fastness assessment at the time of compilation, it has been necessary in this Edition to make provision for the inclusion of data according to one or more of the following—

★

A — (American) The methods of The American Association of Textile Chemists and Colorists.
B — (British) The methods of The Society of Dyers and Colourists.
C — (Continental) The methods of The Deutsche Echtheitskommission.

An important feature of PART I is the sub-division of each usage group into hue groups — Yellow, Orange, Red, Violet, Blue, Green, Brown and Black. The hue classification has been made by reference to a Hue Indication Chart which has been designed specially for the purpose. A copy of the chart is included with each set of volumes.

★ *There is now an accepted international system*

VOLUMES 3 & 4 WILL BE PUBLISHED IN 1957

THE ENLARGED WORK IS IN FOUR VOLUMES

Part 1 is in 2 volumes, Part 2 in 1 volume and Part 3 in 1 volume

ORDERS PLACED NOW will be included in the RELEASE PRICE OF £35 PER SET OF FOUR VOLUMES which will be effective until the end of June 1957 when the price may have to be advanced

Full particulars from

THE SOCIETY OF DYERS AND COLOURISTS

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BENZADONE

NAVY BLUE R

For the production of navy blue shades of very good fastness to light and washing. Excellent for machine dyeing and printing

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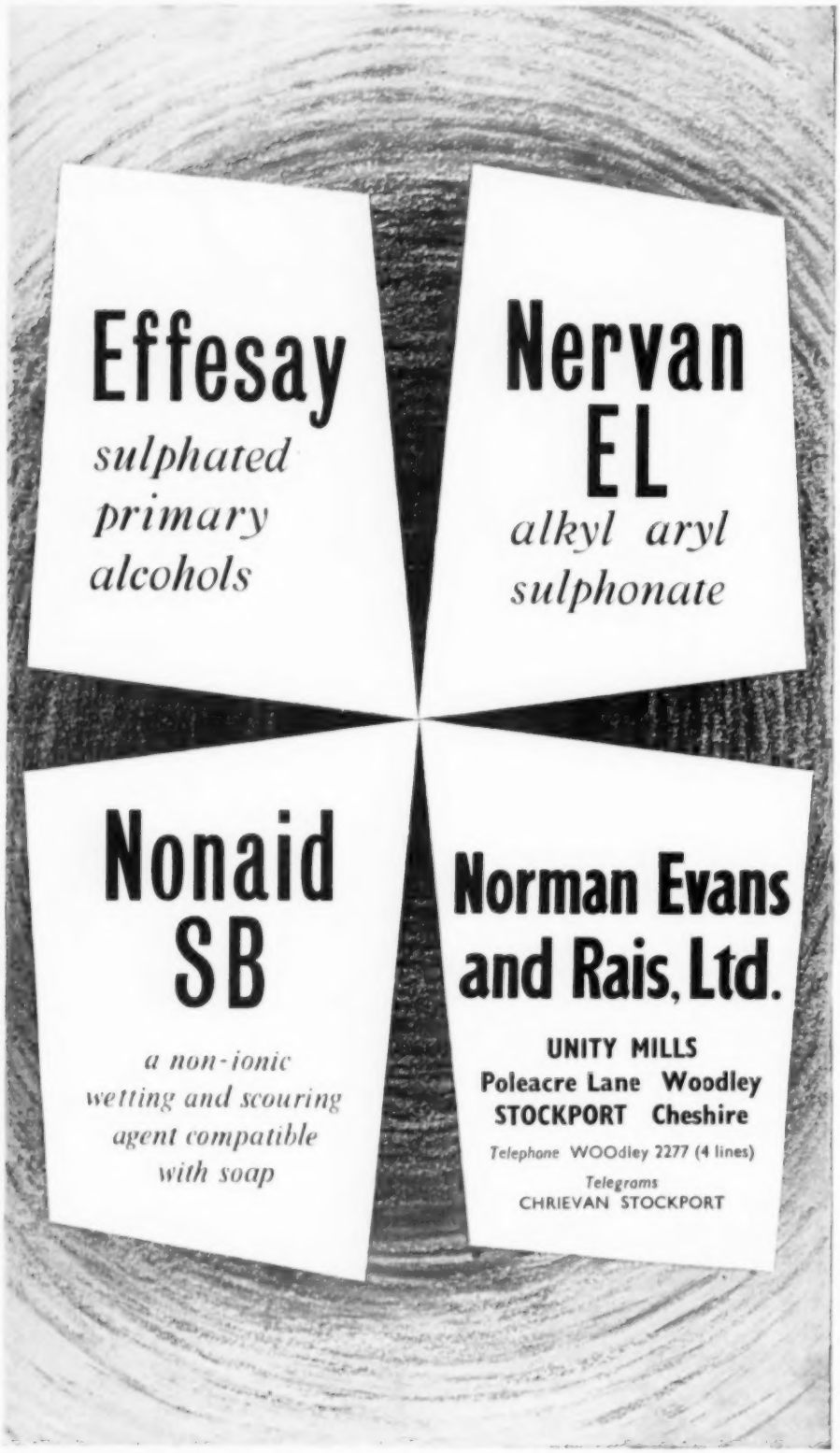
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Considerable saving in time with more level results in the dyeing of fast to milling acid colours and chrome colours.

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DRAYTON REGULATOR & INSTRUMENT CO. LTD., WEST DRAYTON, MIDDLESEX. *Tel.: West Drayton 2611*

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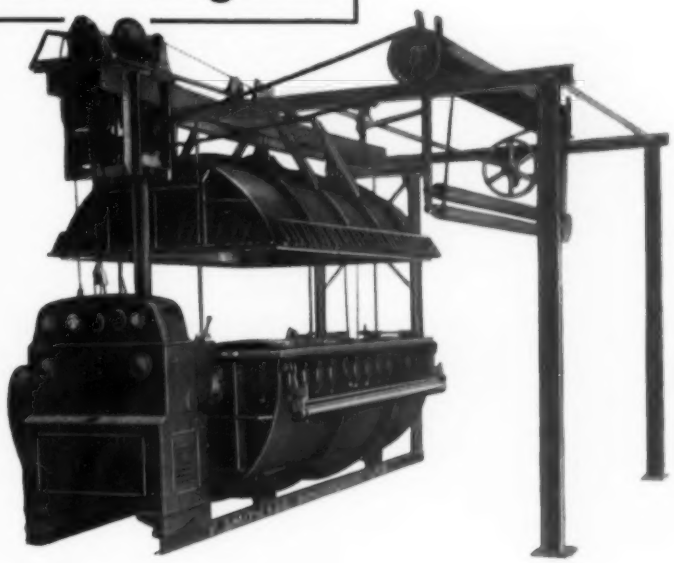

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keeps you up-to-date

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PRESSURE DYEING MACHINES
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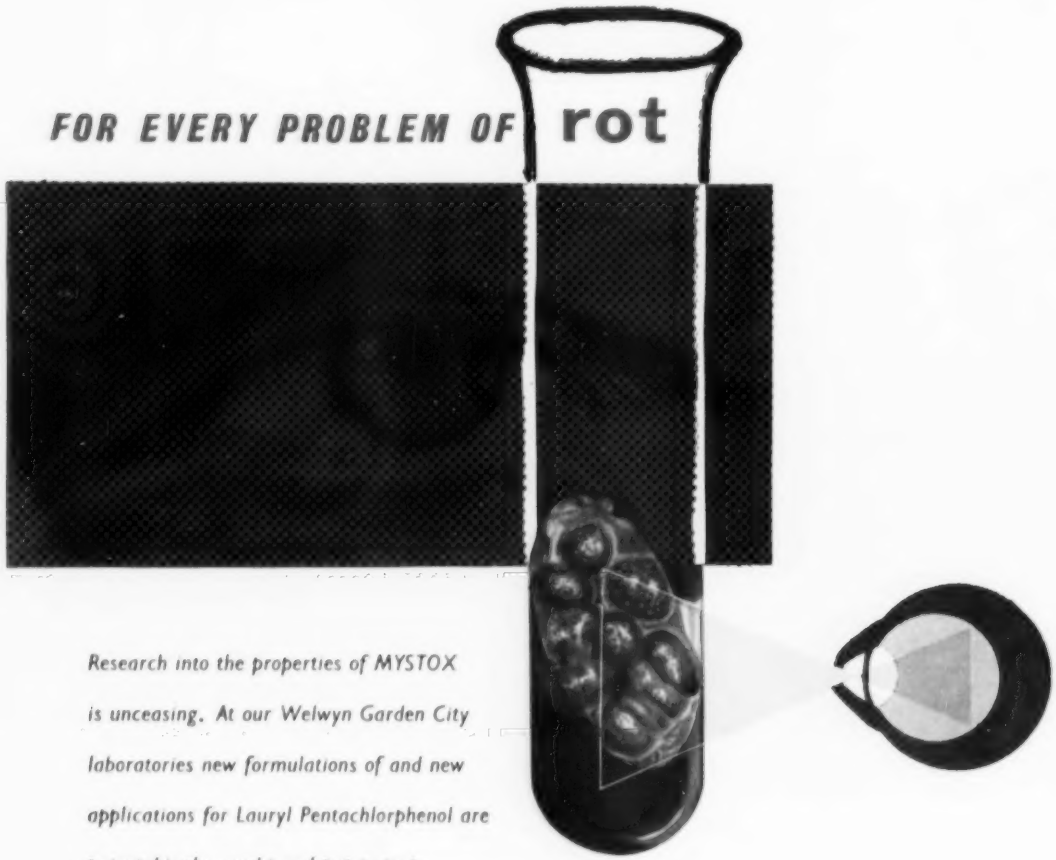

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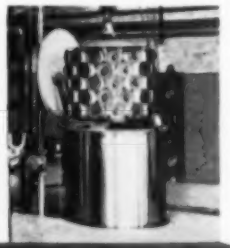

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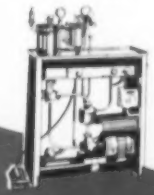
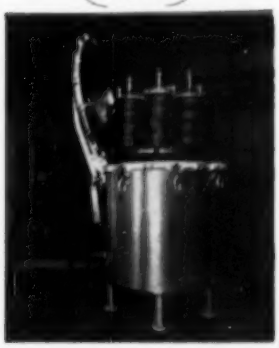
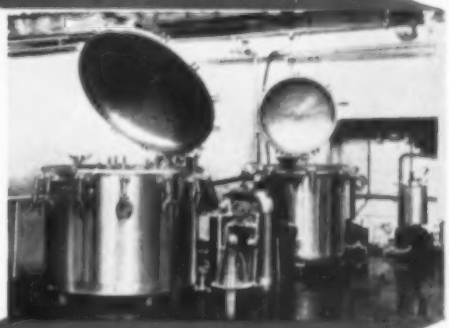


Drying

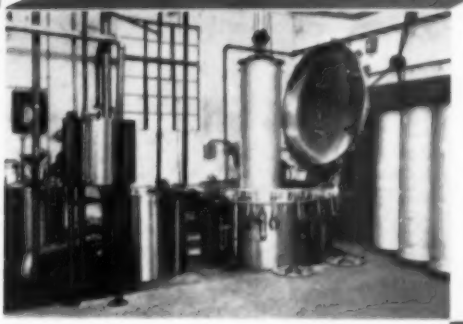


Tops



Pre and after Treatment

Beams



Laboratory
(SKEIN)

Loose Stock

PEGG

PRESSURE DYEING AND DRYING MACHINES

for all types of fibres in every stage of manufacture

TEMPERATURES UP TO 130° C.

Built within the Bentley Group

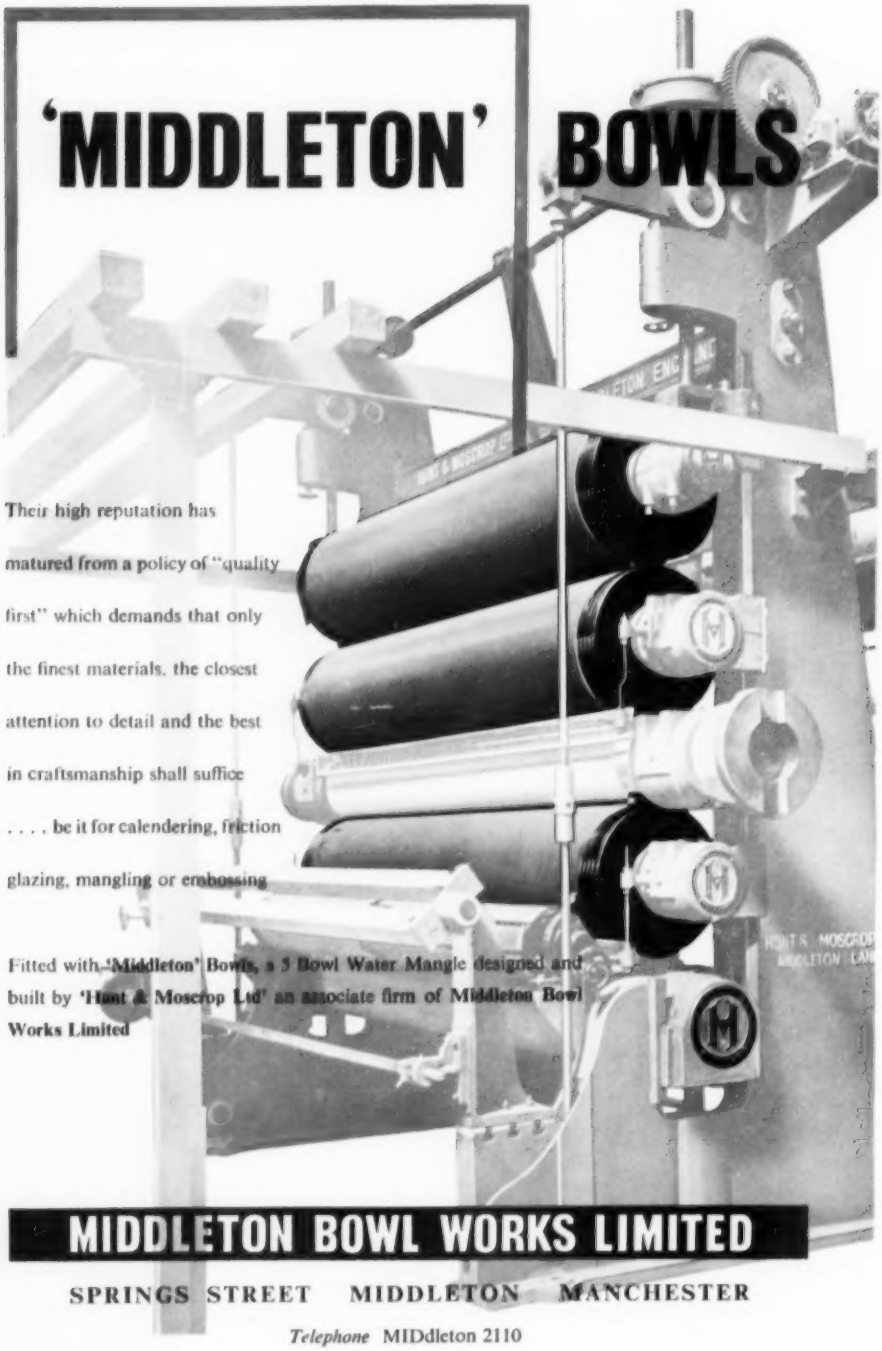
SAMUEL PEGG & SON LTD.

Barkby Road · Leicester · England.

TELEPHONE 66651 TELEGRAMS PULSATOR

SP 47

'MIDDLETON' BOWLS



Their high reputation has matured from a policy of "quality first" which demands that only the finest materials, the closest attention to detail and the best in craftsmanship shall suffice . . . . be it for calendering, friction glazing, mangling or embossing

Fitted with 'Middleton' Bowls, a 5 Bowl Water Mangle designed and built by 'Hunt & Moscrop Ltd' an associate firm of Middleton Bowl Works Limited

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Printing is done on conventional equipment, at top machine speed, usually with a reduction in steaming time and a definite increase in efficiency. Prints show a dyed effect in blotch areas, with excellent overall coverage and smoothness. Delicate patterns are exceptionally sharp.

The SAVAGRAPH process, with VANTASOL dyes, can be used on **any** and **all** cellulosic fabrics. In all cases it offers increased crock fastness over vats printed by present methods, and the **highest degree of fastness** to all color-destroying influences - washing, bleaches, sunlight, and salt water.

If you are now printing with vat dyes and utilizing a flash ager, you too can adapt the SAVAGRAPH process in your plant. Our distributors of Du Pont Dyes will be glad to collaborate in a trial. Get in touch with them!

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Better Things for Better Living
. . . through Chemistry

* Trademark

RANGES OF REPUTATION

DURAMINE and **EVRON FAST**
ACID DYESTUFFS

AZONINE and **DURANTINE FAST**
DIRECT DYESTUFFS

Town End Chemical Works Limited

BRAMLEY · LEEDS 13

analyst." If one assumes that it is the results of applying these tools which are to be interpreted, it may be said that the objective is reached.

In short, this is a book which, in spite of its shortcomings, should find a place on the shelf of the organic analyst.

A. JOHNSON

New Books received

Synthetic Polypeptides—Preparation, Structure, and Properties. C. H. BAMFORD, A. ELLIOTT, and W. E. HANBY. New York: Academic Press Inc. 1956. Pp. xiii + 445. $10.00.

Modern Instruments in Chemical Analysis. Frank M. BIFFEN and William SEAMAN. New York and London: McGraw-Hill Book Co. 1956. Pp. ix + 333. 56s. 6d.

Polyesters and their Applications. BJORKSTEN RESEARCH LABORATORIES INC. New York: Reinhold Publishing Corpn. 1956. Pp. viii + 618. 80s. 0d.

Fünf Kapitel Farbenlehre. Eberhard BUCHWALD. Mosbach, Germany: Physik Verlag. 1955. Pp. 144.

A Handbook of Textile Finishing. A. J. HALL. London: National Trade Press Ltd. 2nd edition 1957. Pp. vii + 250. 25s. 0d.

Experimentelle und theoretische Grundlagen der Trocknung. A. W. LYKOW. Berlin: VEB Verlag Technik. 1955. Pp. 479.

Handbuch für die textile Nassveredlung. Gerhard MELZER. Berlin: VEB Verlag Technik. 1956. Pp. 394. DM 23.00.

Organic Analysis. Volume III. Edited by John MITCHELL *et al.* New York: Interscience Publishers Inc. 1956. Pp. viii + 546. $11.50.

The Chemical Industry in Europe. Paris: O.E.E.C. 1956. Pp. 211. 14s. 0d.

Reports on the Progress of Applied Chemistry. Volume 40 (1955). London: Society of Chemical Industry. 1956. Pp. 1000.

Synthetic Organic Chemicals. UNITED STATES TARIFF COMMISSION. Washington: United States Government Printing Office. 1956. Pp. viii + 187. 65 cents.

Technique of Organic Chemistry. Volume III. Edited by Arnold WEISSBERGER. New York: Interscience Publishers Inc. 2nd edition 1956. Pp. ix + 873. $17.50.

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes.
Any publication abstracted may be referred to by members of the Society on application to
Dr. C. B. Stevens, Dyeing Department, Leeds University

Cassella Farbwerke Mainkur

IMMEDIAL FAST BRILLIANT GREEN 10GL—This sulphur dye gives greens of outstanding brightness for this class of dye, of good fastness to wet treatments, and of comparatively good fastness to chlorine. It has good solubility and is recommended for use alone and in mixtures on all forms of cellulosic material. Reoxidation after dyeing is slow, and treatment in weak acetic acid or potassium dichromate–acetic acid is recommended. Aftertreatment of a dyeing with potassium dichromate–copper sulphate increases its light fastness by one unit, while aftertreatment with Solidogen FFL increases the fastness to washing. Fastness figures on cotton include—Light 5–6, washing (95°C.) 4–5, chlorine 3–4.

INDANTHREN YELLOW GREEN GC—This vat dye gives yellowish greens of excellent fastness to light and wet treatments on all types of cellulosic materials. It does not promote degradation of the dyed material on exposure to light and is thus very suitable for use alone and in mixtures for curtains and furnishings. It is also suitable for dyeing yarns subsequently to be piece-bleached. The Indanthren fastness grading may be given to dyeings on awnings in full depths and on the other categories in any depth. Brands suitable for pigmentation methods are available. Pale to medium depths may be discharged to a pure white. It is of interest for fast-to-washing styles on silk but not on materials which are to be continuously exposed to sunlight. Fastness figures on cotton include—Light 7–8, soda boiling 4 (with Ludigol 5), chlorine 4–5.

MACHINE AND SCREEN PRINTS WITH INDANTHREN GREEN GC—Examples and working details are given of a machine print on cotton and spun viscose rayon staple cloth and of a screen print on spun rayon cloth produced according to the Colloresin process. Fastness figures for prints on spun viscose rayon include—Light 8, washing (95°C.) 5, chlorine 5.

Ciba Ltd.

CIBALAN BLACK BGL—This neutral-dyeing metal-complex dye gives neutral greys and blacks on wool, silk, and polyamide fibres. It may be applied by conventional methods, by a shorter method in which dyeing is begun near the boil, or by the high-temperature pressure method. For full blacks ammonium sulphate can be replaced by ammonium acetate or acid sodium pyrophosphate; free acids should not be used when dyeing wool, but are permissible with polyamide fibres and silk. It is claimed that wool quality is preserved and maximum spinning yield is obtained. The fastness characteristics of dyeings are of the same order as those of other members of the Cibalan range, and, in particular, no yellowish staining on white wool occurs during alkaline milling. Fastness figures on wool include—Light 7–8, alkaline milling (*b*) 4–5, potting 4.

CIBALAN BLACK BGL IN PRINTING—Neutral grey to black direct prints are readily obtainable on wool, silk, and polyamide fibres, only a short steaming being required for fixation. It is also of interest as an outline black in both direct and discharge printing on wool and silk. Fastness figures for a direct grey print on wool include—Light 6, washing (*b*) 5, perspiration 5.

CIBALAN BRILLIANT RED BL—This neutral-dyeing acid dye gives bright bluish reds on wool, silk, and polyamide fibres. Its dyeing characteristics and fastness properties are comparable with those of the Cibalan range, with which it is designed to be used for brightening and shading, being the first red of the Cibalan Brilliant series. A notable feature of this dye is its ability to give solid dyeings on wool–polyamide fibre mixtures. Fastness figures on wool include—Light 6, washing 4 (bluer), alkaline milling (*b*) 5.

CHLORANTINE FAST TURQUOISE BLUE GRLL—This direct dye gives very bright turquoise blues on cellulosic fibres, somewhat redder than are obtained with the GLL brand. It is a readily soluble, very level-dyeing dye, and is suitable for application on the jig and the winch, in circulating-liquor machines, and by high-temperature pressure methods. It gives level dyeings on barry viscose rayon. Since it has low affinity for cellulosic materials, double the normal amount of salt is recommended when dyeing medium and heavy depths, and it is advantageous to add Lyofix SB conc. or Lyofix EW to the first rinse water. An additional aftertreatment with either of these products or with Coprantex B is recommended to improve the wet fastness properties of the dyeing. Fastness figures on viscose rayon include—Light 6–7, perspiration (aftertreated with Lyofix EW) 4–5, washing (*a*) (aftertreated with Lyofix EW) 4 (redder).

CIBANEUTRENE DYES—These are stabilised azoic compositions which can be fully developed by a short, neutral steaming without special additions to the print paste. They give prints of equal brightness and tinctorial strength to the Cibanogene dyes. In addition to being suitable for application alongside vat, direct, etc. dyes, since they can be developed very satisfactorily by dry heat they can be printed alongside the Oremasin resin-bonded pigments. They give bright-coloured resists under Cibantines and Aniline Black, and like the Cibanogenes can also be

developed in acid steam or in a hot acid bath. Prints of eleven members of the range are included. Fastness-to-light ratings cover the range 3–4 to 6, to washing (*c*) 4–5 to 5, and to chlorine 4 to 5.

Compagnie Française des Matières Colorantes

THE NEUTROGENE VA AZOIC PRINTING COLOURS— This card, issued by the Manchester laboratory of Messrs. Allied Colloids (Bradford) Ltd., contains prints on cotton of twelve azoic printing compositions. These contain improved stabilisers and are superior to the previous Neutrogene range in the following respects— (1) They give print pastes of greatly improved stability. (2) They are more resistant to reduction during steaming. (3) Rather brighter prints are obtained, particularly before soaping. (4) Less caustic soda is required in the print paste, with consequent reduction in risk of damage to silk screens and improvement in the working properties of the print pastes.

THE DYEING OF ACETATE RAYON WITH ACETOQUINONE, DIAZO-ACETOQUINONE, AND ACETAZOL COLOURS— This pattern card contains dyeings on bright and delustred secondary cellulose acetate yarn of 38 disperse dyes, including 3 diazotised-and-developed blacks, a diazotised-and-developed navy, and 2 Acetazol blacks, which only require aftertreatment in a sodium nitrite–formic acid bath to develop the final black. The Acetazol blacks are of particular interest on nylon. The text (47 pp.) contains details of dyeing methods, fastness properties and fastness testing, stripping with Decolorant S and sodium chlorite, and the dyeing of mixtures of cellulose acetate and cellulosic fibres, wool, silk, and nylon.

The Geigy Co. Ltd.

IRGALAN BLUE RL— This neutral-dyeing metal-complex dye gives blues to navies on wool and polyamide fibres, redder and purer in tone than the GL brand, which it closely resembles in dyeing behaviour. The fastness characteristics of dyeings are virtually the same as those of the latter, which are, however, still superior in fastness to chlorine. Fastness figures on wool include— Light 6, washing (Test *b*) 5, heavy milling 5.

IRGALAN BROWN FL— This neutral-dyeing metal-complex dye gives full browns on wool and polyamide fibres. Dyeings appear redder in artificial light, and it is thus very suitable for matching dyeings showing this change, while in combination with Browns 2GL, 2RL, and 3BL, which become greener under these conditions, any desired effect may be obtained. It is also of interest for dyeing silk, on which it gives slightly yellower dyeings, which are readily dischargeable to a good white. Fastness figures on wool include— Light 6, washing (Test *b*) 4, severe milling 4.

IRGALAN RED 2BL—This neutral-dyeing metal-complex dye gives reds intermediate in hue between those obtained with Irgalan Red 3G, which are yellower, and with Rubine RL, which are much bluer. It is of interest on all forms of wool, silk, and polyamide fibres and also for direct prints on chlorinated wool. Fastness figures on wool include— Light 6, washing (Test *b*) 4–5, severe milling 4–5.

DYES FOR WOOL–POLYAMIDE FIBRE MIXTURES— This card contains 63 dyeings on a wool–spun nylon staple mixture produced with dyes selected from the Irgalan, Irganol S, Polar, and other ranges. The dyes used have been divided into four groups: Group A— Dyes which in presence of Erional NW dye both fibres to approximately the same depth and hue. Without restraining agent, the polyamide fibre is dyed more deeply than the wool. Group B— Dyes which in presence of Erional NW dye the wool only. In absence of restraining agent the nylon is slightly coloured. Group C— Dyes which even in presence of Erional EW dye the polyamide much more deeply than the wool. Group D— Dyes for special colours, blues in particular. With Erional NW the wool is dyed much more deeply than the nylon. Without it, the depth on the two fibres is the same, but the hues are different. In addition to a comprehensive range of dyeings illustrating the behaviour of each dye in presence of different amounts of Erional NW there are two sets of mixture dyeings. The first illustrates the production of dyeings of equal depth and of the same hue on both fibres by using combinations of Irgalans as a basis and shading either the wool, the nylon, or both; and the second demonstrates the possibility of obtaining multicoloured effects by a single-bath method using selected dyes in presence of Erional NW. The patterns are composed of samples of 100% wool and spun nylon yarn dyed in the same bath and mounted side by side; fastness data are included for each dye on both fibres, the figures being printed in distinct colours to facilitate reference.

IRGALITE PIGMENTS FOR PRINTING INKS— This pattern card is an extremely lavish production, describing the properties and uses of 50 pigments grouped according to chemical type: (1) Pigmentary colours — insoluble azo compounds containing no base or metallic salts. (2) Double and triple salt complex toners— complexes of basic dyes with phosphomolybdic acid and phosphomolybdotungstic acid respectively. (3) Toners— insoluble barium, calcium, or manganese salts of sparingly soluble dyes. Irgalite Green DBN is an iron complex. A general description of the chemical type of each pigment is given under its name. Six different prints of each are given— full-strength silk-screen, alkyd-resin varnish; full-strength letterpress, linseed stand oil; graduated half-tone, linseed stand oil; alumina 1:5 reduction letterpress, linseed stand oil; titanium dioxide 1:25 reduction silk-screen, alkyd-resin varnish; titanium dioxide 1:25 reduction letterpress, linseed stand oil. Comprehensive fastness data are included together with details of test methods used.

Hickson & Welch Ltd.

INTERMEDIATES: SPECIFICATIONS— This brochure contains specifications of some 36 intermediates for the manufacture of dyes, pharmaceutical products, pigments, and lakes and 10 technical-grade insecticides. Descriptions are also included of five Photines (fluorescent brightening agents), three sulphur blacks, and a range of eight Fast Bases for azoic combinations.

Howards of Ilford Ltd.

SOME APPLICATIONS OF HOWARDS TECHNICAL CHEMICALS— This folder includes the following booklets and leaflets— *Technical Chemicals for the Textile Trade*, *Sextol in the Textile Trade*, cyclo*Hexanol in the Textile Trade*, and *Industrial Soaps, Cleansing Agents, and Textile Oils based on* cyclo*Hexanol and Sextol* (*Methyl*cyclohexanol).

E. I. du Pont de Nemours & Co. Inc.

TECHNICAL BULLETIN, **12**, NO. 1 (MARCH 1956)— In addition to notes on the properties and applications of new products, this issue contains an account of the dyeing behaviour of a new brand of Orlon acrylic fibre which is whiter and absorbs dye more readily, but which in all other respects is identical with type 42 (or 39). In the three years that Orlon 42 has been available its receptivity for cationic dyes has already been increased by 30%. The latest improvement (40% increase) means that jet blacks can now be produced at 205°F. with this class of dye. In consequence, when producing light to medium depths the amount of retarding agent required may have to be increased by 50–75%.

TECHNICAL BULLETIN, **12**, NO. 2 (JUNE 1956)— Articles of special interest include— *Dyeing Blends of Acrilan Acrylic Fibre and Wool*, *Dyeing of Dacron Polyester Fibre using Carriers*, and *Application Methods for Improved Levelness of Dyes on Nylon*.

TECHNICAL BULLETIN, **12**, NO. 3 (SEPTEMBER 1956)— The following articles are of special interest— *Application of Dyes to Wide Carpets by Pad–Steam and Pad–Beck Methods*, *Discharge Printing of Pure Silk*, *Package-dyeing Blends of Dacron Polyester Fibre and Cotton Yarn*, and *Thermosol Dyeings on 100% Spun Dacron Polyester Fibre*. The article on carpet-dyeing describes methods of dyeing rayon carpets with direct and vat dyes using a Niptrol padder (Rodney Hunt Machine Co.) and developing in either the beck or the steamer, and wool and nylon, separately or in blends, with neutral-dyeing metal-complex or acid dyes. The advantages to be gained by using such methods are outlined, and with the rapid growth of the tufted-carpet industry (33% of total yardage produced and amounting to 30 million sq.yd. of rayon carpeting alone) and the importance of the car carpet trade it is considered that batchwise and particularly continuous methods of piece-dyeing carpeting merit serious attention. The article on the Thermosol process consists of fastness data for six disperse dyes on spun Dacron cloth dyed for 1 min. at 200°C.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952) and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

Fires in the Textile-finishing Trade

D. W. Millar and J. F. Fry

J. Textile Inst., **47**, P 879–P 893 (Oct. 1956)

A description of part of the programme of the Joint Fire Research Organisation of the Department of Scientific and Industrial Research. The statistics relating to fires in the finishing trade occurring in the United Kingdom during 1946–1950 are discussed under the following heads—occupancies where fires took place, causes of fires, materials first ignited, points of outbreak, times of outbreaks, losses in fires, types of damage, delays in discovery, and the interrelation of these factors. Use of sprinklers is discussed. Fly, dust, and fluff are shown to be serious causes of fires; suggestions are made for fire prevention. P.T.S.

Copper and Chromium Plating of Textile-printing Rollers

Anon.

Z. ges. Textilind., **58**, 883–886 (20 Nov. 1956)

Details are given. Chromium-plated rollers have a harder surface than copper-plated rollers and are therefore more resistant to mechanical damage. B.K.

Cutting Mechanisms of Shearing Machines

R. Schmidt

Tintorería industrial, (19), 453–468 (Sept. 1956)

The elements composing the cutting mechanism are discussed in turn and details with illustrations are given of types of spirals, adjustments to and grinding of the cylinder, blades of various hardness, and methods of sharpening them. S.R.C.

PATENTS

Continuous Treatment of Threads in Apparatus of J-Box Type

Société Rhodiacéta *BP* 763,364

Wet Treatment of Thread

Courtaulds *BP* 763,471

The thread is led over a stationary sloping surface down which the treating liquor flows and whose upper surface scrapes the liquid from the surface of a rotating horizontal roller to which the liquid is supplied. C.O.C.

Apparatus for Continuously Applying a Colouring Fluid to a Thread, Wire or the like

Western Electric Co. *USP* 2,726,631

Drying Leather

Secoterm *BP* 763,573–4

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Table of Textile Auxiliaries

St. Jost

Textil-Rund., **11**, 723–736 (Dec. 1956); **12**, 20–23 (Jan. 1957)

Information collected from the *American Dyestuff Reporter* and the *Deutscher Färberkalender* with additions. Some 360 auxiliaries are covered. S.R.C.

Industrial and Medical Uses of Carbohydrates

M. Stacey

Chem. and Ind., 1398–1408 (1 Dec. 1956)

A brief review. W.R.M.

Hydrosulphite and Rongalite (R) after Fifty Years

A. Janson and H. A. Nelson

Amer. Dyestuff Rep., **45**, 797–799 (22 Oct. 1956)

See J.S.D.C., **72**, 541 (Nov. 1956).

Aromatic Reactivity. I—Effects of Substituents on the Acid Cleavage of Phenyltrimethylsilanes

C. Eaborn

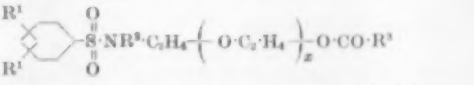
J.C.S., 4858–4864 (Dec. 1956)

PATENTS

Textile Lubricants and Antistatic Agents

Monsanto *USP* 2,727,004

Compounds of formula—



(R^1 = H, CH_3 or C_2H_5; R^2 = H, CH_3, C_2H_5, $-C_2H_4OH$,

$-C_2H_4-(-O\cdot C_2H_4-)_y-OH$ or $-C_2H_4-(-O\cdot C_2H_4-)_y-O\cdot CO\cdot R^3$;

R^3 = sat. or unsat. aliphatic hydrocarbon of 5–23 C; x = 1–20; y = 1–19; $x + y < 21$) are used, particularly on synthetic fibres and in association with mineral oil, as lubricants and antistatic agents. C.O.C.

Lubricants for Cellulose Ester Fibres

Celanese Corpn. of America *USP* 2,727,860

A blend of a mineral oil, an alkyl phenol, a vegetable oil lower aliphatic ester, a modified vegetable oil, a higher fatty acid, an amine and an alkyl phosphate, is a textile lubricant having antistatic properties. C.O.C.

Polymeric Surface Active Agents

American Cyanamid Co. *BP* 764,409

Copolymers of acrylamide or methylacrylamide and an acrylic or methacrylic ester of a saturated aliphatic alcohol of 8–22 C, e.g. *n*-lauryl methacrylate, are excellent wetting and emulsifying agents. C.O.C.

Sodium *p*-Toluenesulphonbromamide

H. Wallrath *BP* 763,383

Sodium *p*-toluenesulphonbromamide in aqueous solution liberates Br faster than Cl is liberated by the corresponding chlorine compound and is therefore a better disinfectant for incorporation into detergents. C.O.C.

Emulsifying Agents

FBy *BP* 763,396

The products obtained by agitating an aq. soln. of an alkali metal hydroxide with a solution in a water-immiscible organic solvent of a copolymer of a water-insoluble vinyl compound with one of formula—

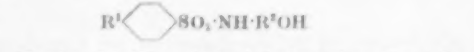
$$R^1\cdot CH{:}CX\cdot COOH$$

(X = H, Hal or Alk; R^1 = $COOR^2$, $CONHR^2$ or $CONR^2R^3$ (R^2 and R^3 = Alk of > 8 C)) are useful emulsifying agents. Those made from monoesters of ethylene-$\alpha\beta$-dicarboxylic acids with fatty alcohols of > 8 C are particularly suitable. Used in paints, water-repellent finishes for textiles or leather finishes they have the advantage that they do not affect the water-repellent property of the film or finish. C.O.C.

Emulsifying and Dispersing Agents

Boehme Fettchemie *BP* 763,360

Compounds of formula—



(R^1 = Alk of > 5 C; R^2 = aliphatic hydrocarbon of > 1 C and which may carry additional OH groups or be interrupted by an O atom) are good agents for producing aqueous emulsions, e.g. R^2 = $CH_2{\cdot}CH_2$, $CH_2CH(OH)CH_2$ or $CH_2CH_2O{\cdot}CH_2CH_2$. C.O.C.

Dispersions Stable to Electrolytes

Boehme Fettchemie *BP* 766,177

Aqueous dispersions formed by use as dispersing agent of condensation products from polyalkylene oxide waxes, oxo-compounds and higher molecular aliphatic, *cyclo*-aliphatic, aliphatic-*cyclo*aliphatic or aliphatic aromatic compounds which contain a replaceable H atom linked through O, S or N, are stable toward electrolytes. C.O.C.

Stabilisers for Per Compounds in Detergents

Thomas Hedley & Co. *BP* 765,750

Ethylene diamine tetraacetic acid and β-hydroxyethyl ethylene diamine triacetic acid and their salts are excellent stabilisers for per compounds contained in synthetic detergent compositions. C.O.C.

Detergent Compositions for Removing Radioactive Contamination
United Kingdom Atomic Energy Authority
BP 763,547

Addition Compounds of Amines and Halides of Aluminium and Boron—Detergents, Wetting, Emulsifying and Rotproofing Agents
BP 763,455

Compounds of formula—

$$(R_2N)_nEX_3 \text{ or } [(R_2N)_nEX_{4-n}]\cdot(n-1)X$$

(only one of the R's is attached to the N atom by a terminal C atom and is a straight chain Alk, alkenyl or alkadienyl of 12–18 C; the other Rs = H, Alk of 1–5 C, *cyclo*hexyl, phenyl or benzyl; E = Al or B; X = Cl, Br or I; n = 1–4, when n = 1–3 X may be F provide E is B), e.g. a complex of monolaurylamine (4 mol.) and BCl_3 (1 mol.) of melting range 148–154°C. have widely varied uses as auxiliary and finishing agents. C.O.C.

Antistatic Agents
Hoechst Fettchemie *BP* 763,215

Alkylene oxide addition products of organic sulphamides having a hydrocarbon radical of > 5 C, e.g. the addition product of tri*iso*propylbenzene-*N*-hydroxyethylsulphamide with 9 mol. ethylene oxide, are antistatic agents which also impart a soft handle.

BP 763,216

Alkylene oxide addition products of organic carboxylic acid monoalkanolamides, e.g. the addition products of stearic acid monoethanolamide with 4 mol. ethylene oxide, preferably together with a wax or high mol.wt. fatty or wax alcohol, are used. C.O.C.

Emulsified Hydrogen Peroxide
Petrochemicals *BP* 763,496

Very stable emulsions of hydrogen peroxide are obtained by mixing together aqueous hydrogen peroxide and a liquid aromatic hydrocarbon containing no unsaturated aliphatic groups and boiling within 100–300°C. containing in solution a non-ionic surface active compound. C.O.C.

Pigments and Finishing Agents (XII p. 118)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

In the Footsteps of Perkin
W. H. Cliffe
J.S.D.C., **72**, 563–566 (Dec. 1956)

It is less easy to make Perkin's Mauve than may be supposed. With little more than the original patent specification as a guide, yet with all the facilities of modern laboratories, chemists of 1956 have experienced difficulty in preparing on a small scale Mauve equal in quality to that which Perkin & Sons manufactured almost a century ago. AUTHOR

Some Early Stages in the Renaissance of the British Dyemaking Industry—Tales from Turnbridge, Huddersfield, 1899–1920
C. M. Whittaker
J.S.D.C., **72**, 557–563 (Dec. 1956)

Celebration of the Centenary of the Discovery of Mauve by W. H. Perkin
C. J. W. Hooper
J.S.D.C., **72**, 566–573 (Dec. 1956)

The various methods adopted to commemorate in 1956 the centenary of the discovery by William Henry Perkin of Mauve, the first coal-tar dye to be manufactured and applied on a large scale, are outlined, and a bibliography is given of the most important articles which have appeared. AUTHOR

Ultraviolet Absorption of Isolated Double Bonds
O. H. Wheeler and J. L. Mateos
J. Org. Chem., **21**, 1110–1112 (Oct. 1956)

Energy Levels and Absorption of some Mono-substituted Benzene Derivatives
A. Mangini and C. Zauli
J.C.S., 4960–4967 (Dec. 1956)

Separation of Arylsulphonic Acids
H. Feilchenfeld
Ind. Eng. Chem., **48**, 1935–1937 (Oct. 1956)

Ternary phase diagrams from which optimum separation conditions can be calculated, are presented for the systems sulphuric acid–water with (*a*) *p*-toluenesulphonic acid (30°, 45°, 60°C.), (*b*) *p*-xylenesulphonic acid (30°, 60°C.), and (*c*) the sulphonic acids prepared from petroleum naphtha (b.p. 130°–170°C.). In each case, maximum separation occurs at a spent-acid composition of approx. 65% sulphuric acid, 35% water. W.K.R.

Preparation of *p*-Toluenesulphonyl Chloride
T. Ognyanov and A. Zagorova
J. Appl. Chem. U.S.S.R., **29**, 1299–1301 (Aug. 1956)

A 77% yield of *p*-toluenesulphonyl chloride is obtained by treating the toluene with 4–5 mol. of chlorosulphonic acid at 60–70°C. in the presence of 0·5 mol. of Na_2SO_4. The reaction carried out at 30°C. was found to favour the formation of the *ortho* isomer. Good yields were also obtained using $FeCl_3$, $ZnCl_2$, or $SnCl_2$ instead of Na_2SO_4. The best (nearly 100%) yield was obtained in the presence of $ZnCl_2$. T.Z.W.

Reactions with Nitrosodisulphonate. XIV—Substitution Products in the Naphthalene Series
H.-J. Teuber and N. Götz
Chem. Ber., **89**, 2654–2669 (Nov. 1956)

Manufacture of Phthalic Anhydride by the Fluidised-catalyst Method
H. L. Riley
Chem. and Ind., 1464–1468 (15 Dec. 1956)

The spectacular increase in the production of phthalic anhydride in the U.S.A. is described and its present uses are briefly reviewed. It is then stated that prior to 1944 all the phthalic anhydride produced by air oxidation was manufactured in fixed-bed reactors, after which date the first fluidised-catalyst unit was operated by the Sherwin-Williams Co. of U.S.A. Advantages claimed for the later process are—(i) operation at uniform and easily controlled temp. with resultant purer product, (ii) use of lower air : naphthalene ratios, (iii) elimination of explosion dangers, (iv) simpler condensation, and (v) utilisation of heat of reaction. The development of the fluidised-catalyst method for large-scale production is described, and the difficult design and operational problems involved are discussed in detail. In particular, the contributions made by the Rotherwood Laboratories (the research and development department of the United Coke & Chemicals Co. Ltd.) are given at length, and include solution of the problems of filtration (such as loss of catalyst dust) by the use of glass fibres, and of condensation, in which operational troubles due to corrosion, leakages, blockages, dirty heat-exchange surfaces, etc. can be rectified without a major shut-down of the plant. It is concluded that the system should use the type of heat-exchangers employed in air conditioning. The new method affords a product substantially free from both maleic anhydride and naphthaquinone, in yields appreciably higher than those reported for fixed-bed plants. 11 References and 5 illustrations. H.H.H.

Preparation of *o*-Dinitrobenzene
Y. Ogata and M. Tsuchida
J. Org. Chem., **21**, 1065–1066 (Oct. 1956)

o-Dinitrobenzene is obtained in 91% yield from *o*-nitrophenylmercuric chloride on reaction with HNO_3 at 95°C. for 10 min. A 26% yield together with 24% of the *meta* isomer is recorded for the nitration of nitrobenzene in presence of HgO at 95°C. for 10 hr. Extension of the mercuric-ion-catalysed procedure to the synthesis of unusual cpd. proved unsuccessful. H.E.N.

Steric Inhibition of Mesomerism in Alkyl Derivatives of 1:3:5-Trinitrobenzene
E. Liss and K. Lohmann
Chem. Ber., **89**, 2546–2550 (Nov. 1956)

Reduction of Nitrobenzene and its Derivatives VII—Action of Lime in Reduction using Ferrosilicon
H. Iida
Rept. Govt. Chem. Ind. Research Inst. Tokyo, **51**, 385–391 (Oct. 1956)

In the preparation of hydrazobenzene by the reduction of nitrobenzene in caustic soda solution with ferrosilicon as reducing agent, the addition of lime to the reaction mixture facilitates the smooth reduction even at so low a caustic soda concn. as 15%. The rôle of lime in this reaction is studied.

VIII— Polarographic Study of Alkaline Reduction of Nitrobenzene, etc.

H. Iida and K. Kayahara

Ibid., 392–396

When nitrobenzene is treated in alkaline solution in the presence of finely powdered zinc or ferrosilicon, it can be reduced step by step through azoxybenzene and azobenzene to hydrazobenzene. Therefore, it follows that the order of the ease of reduction of these compounds under such experimental conditions is: nitrobenzene > azoxybenzene > azobenzene > hydrazobenzene. However, published data on the polarographic half-wave potentials of these compounds in solutions of pH 3–12 indicate that azobenzene is more reducible than azoxybenzene and nitrobenzene. In other words, the published polarographic data do not appear to be in accordance with the actual course of the reduction of nitrobenzene in alkaline solution with zinc or ferrosilicon. Moreover, the half-wave potentials of the compounds in solutions more alkaline than pH 12 are not found in the available published literature. The present polarographic experiments confirm that the half-wave potentials of the nitro and azoxy compounds in solutions more alkaline than pH 14 are more positive than those in solutions of pH 12, whereas the potentials of azo compounds become more negative with increasing pH value. Further, it is confirmed that the experimental facts observed in the reduction of nitrobenzene in alkaline solution with zinc or ferrosilicon can be explained by the order of the half-wave potentials of nitrobenzene, azoxybenzene, and azobenzene in 3*N*-NaOH at 45°C. AUTHORS

Oxidation Products of Phloroglucinol

H. N. Stein and H. J. C. Tendeloo

Rec. Trav. chim., **75**, 1233–1239 (Sept.–Oct. 1956)

Benzene-soluble Aryldiazonium Salts

W. Bradley and J. D. Thompson

Nature, **178**, 1069–1070 (10 Nov. 1956)

Unlike previously known diazonium salts, the following are moderately sol. in warm benzene (but sparingly so in the cold)— *p*-*n*-decyloxybenzenediazonium toluene-*p*-sulphonate, *p*-cetyloxybenzenediazonium chloride, and 2:5-di-*n*-decyloxybenzenediazonium chloride. They appear to exist in benzene as ion pairs, though more complex association cpd. of molecules and ions may also be present. These salts react readily with *β*-naphthol in the same solvent, and in presence of a trace of pyridine, to give normal azo compounds; they also react freely with typical Grignard reagents. J.W.D.

Metal Chelate Compounds of Azophenols

N. Kanemiwa

I— Copper Complex of Phenylazo-*p*-cresols and Phenylazoresorcinols

J. Pharm. Soc. Japan, **76**, 787–792 (1956): *Chem. Abs.*, **50**, 16518 (25 Nov. 1956)

Spectroscopic study of the formation of a complex salt between Cu^{2+} and 17 compounds including *p*-HO·C_6H_4·N:N·C_6H_3, 4:2-$CH_3(C_6H_5$·N:N)C_6H_2·OH (I), 4:1:3-C_6H_5·N:N·$C_6H_2(OH)_2$ (II), 2-OH·$C_{10}H_6$·N:N·C_6H_4·OH-2′ (III), 2-HO·C_6H_4·N:N·C_6H_4·OH-2′ (IV), and 4:1:3-(*o*-HO·C_6H_4·N:N)$C_6H_3(OH)_2$ (V) showed fairly distinct ability to form a complex salt using $1{\cdot}67 \times 10^{-5}$ M. soln. in 50% ethyl alcohol and heating for 30 min. at 100°C. Formation of a complex becomes more difficult in the order III, IV, V, II, and I; the benzene rings containing CH_3·O, NO_2, and other substituents also showed some difference in complex-salt formation. The effect on complex formation of various substituents in compounds of the azophenol series is discussed.

II— Metal Chelate Compounds of *oo*′-Dihydroxyazo Compounds

Ibid., 972–974

Examination of the formation of complexes of III, IV, and V with Cu, Co, Ni, Mg, Zn, Cd, and Mn showed that they formed complex salts readily in the order Cu > Co, but only V formed complexes with Ni, Mg, and Zn. Cd and Mn did not form complexes. The compositions of some of these complex salts and that of Cu complexes of II were determined by Job's method.

III— Metal Chelate Compounds of Azophenols in Strong Alkaline Solution

Ibid., 795–798

Formation of complex salts in 0·5*N*-NaOH was examined spectrophotometrically with 10 azophenols. IV and V form complexes with Cu, Co, and Ni, but not with Zn, Cd, or Mn. The concentration of the dye and inorganic ions was $1{\cdot}67 \times 10^{-6}$M. Some of the dyes decomposed in the alkali, this decomposition being accelerated by Cu^{2+}. C.O.C.

Furan Dyes— Diacetarylides of Furan-2:5-dicarboxylic Acid as Azoic Coupling Components

G. Modena

Boll. sci. Fac. Chim. ind. Bologna, **14**, 17–21 (1956): *Chem. Abs.*, **50**, 16740 (25 Nov. 1956)

Yellow azoic dyes applied to cotton by padding with various furan-2:5-dicarbonylacetarylides and coupling with suitable diazo compounds were subjected to some fastness tests. The coupling components were made by condensing furan-2:5-dicarbonyl chloride with ethyl acetoacetate in benzene in presence of aq. NaOH, and treating the diethyl furan-2:5-dicarbonylacetate so formed in xylene with a primary aromatic amine (e.g. *o*-anisidine) in presence of a trace of piperidine. E.S.

Interaction between Solid Dye Layers and Water Vapour at Temperatures about 100°C.

A. T. Vartanyan

Zhur. fiz. khim., **30**, 424–436 (1956): *Chem. Abs.*, **50**, 16383 (25 Nov. 1956)

The absorption spectra of Methylene Blue and Crystal Violet obtained by vacuum sublimation are similar to those of their concentrated aqueous solutions, while the short-wave absorption spectra at 540 mμ. of an aqueous solution of Crystal Violet and of the layer obtained by precipitation from the solution are of different origins. Rhodamine B and G become colourless by thermal activation during vacuum sublimation, and have characteristic absorption bands at 318, 277, and 235 mμ. They are converted back to the coloured dye immediately they are dissolved. Rhodamine 6G is unchanged by vacuum sublimation. Methylene Blue and Crystal Violet form leuco compounds with water vapour above 100°C. C.O.C.

Mechanism of Dye Formation in Colour Photography. II— Salt Effects on Deamination Rate of Oxidised *p*-Phenylenediamines

L. K. J. Tong and M. C. Glesman

J. Amer. Chem. Soc., **78**, 5827–5829 (20 Nov. 1956)

The most probable ionic forms of the quinonediimine resulting from the oxidation of *NN*-disubstituted *p*-phenylenediamine have been derived from measurement of the salt effects on the rate of deamination of the quinonediimines in alkaline solution. C.O.C.

Copper Complexes of *oo*′-Dihydroxy Schiff's Bases

C. Dikmen and T. Gündüz

Chem. Ber., **89**, 2637–2641 (Nov. 1956)

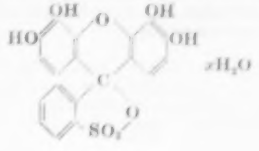
Such 1 : 1 complexes are prepared from the Schiff's bases obtained from salicylaldehyde with *o*-hydroxybenzylamine (I) or 1-aminomethyl-2-naphthol (II) and from 2-hydroxy-1-naphthaldehyde with I and with II. The first and third are green, the others brown. They are stable to boiling 2*N*-NaOH, but not to hot 2*N*-HCl. They can co-ordinate 1 mol. of amine or pyridine. H.E.N.

Sulphonephthaleins. II— Preparation and Properties of Pyrogallolsulphonephthalein, "Pyrogallol Red"

Z. Vodák and O. Leminger

Chem. Listy, **50**, 943–947 (1956): *Chem. Abs.*, **50**, 16717 (25 Nov. 1956)

o-Sulphobenzoic anhydride (276 g.), pyrogallol (378 g.), and dry xylene (480 ml.) are heated, the water formed being continuously removed. The xylene is then removed by steam distillation, and the crystalline suspension washed with hot water. The crystals are dissolved in aq. Na_2CO_3, filtered, and precipitated with 6*N*-HCl to yield 360 g. of crystals of pyrogallolsulphonephthalein,H_2O. It is obtained in the anhydrous form by boiling for 18 hr. in xylene; it forms the monohydrate again immediately water is added. Both the anhydrous and non-hydrate forms yield a fibrous polyhydrate form when they are dissolved in aq. alkali and then precipitated with acid. The absorption spectra of the three forms suggest the formula—

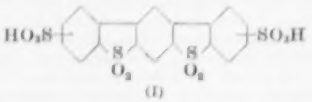


It forms intensely coloured complexes with many cations, e.g. in alkaline soln. Mg^{2+}, Cd^{2+} (violet), Co^{2+} (bluish violet), Ni^{2+} (blue); in weakly acid soln. Cu^{2+}, Pr^{3+}, Nd^{3+}, Pb^{2+}, UO^{2+} (violet), Mo^{VI}, W^{VI} (reddish violet); in strongly acid soln. Bi^{3+} (red). C.O.C.

Phenazine Syntheses. IX— 1-Halogenophenazines
D. L. Vivian
J. Org. Chem., **21**, 1188–1190 (Oct. 1956)

Formation of Substituted Dibenzothiophen Dioxides by Sulphonation of *m*- and *p*-Terphenyls
J. A. VanAllan
J. Org. Chem., **21**, 1152–1155 (Oct. 1956)

Sulphonation with 20% oleum at 90–100°C. converts *p*-terphenyl into 4′:7-disulpho-3-phenyldibenzothiophen dioxide, *m*-terphenyl into disulpho-10:12-dithiaindeno-[2,1*b*]fluorene 10:12-bisdioxide (I), and quaterphenyl into 3-(4′-sulphodiphenylyl)-7-sulphodibenzothiophen dioxide. Sulphonation by H_2SO_4 at 100°C. of 2-hydroxy-, 2-methoxy-, or 2-ethoxy-benzoyl deriv. of 4′:4″-diamino-*p*-terphenyl occurs in the 5:5′-positions. The 4-methoxybenzoyl deriv. would not sulphonate under these conditions, but would under more vigorous ones, sulphone formation occurring also. All these cpd. are characterised by their exceptionally strong blue–green fluorescence.



(I)

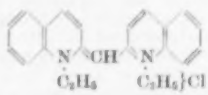
H.E.N.

Stereoisomerism and Excited States of Simple Polymethin Dyes
F. Baumgärtner, E. Günther, and G. Scheibe
Z. Elektrochem., **60**, 570–572 (1956): *Chem. Abs.*, **50**, 16405 (25 Nov. 1956)

1:5-*NN′*-dipyrrolidylpentamethin perchlorate (I) is converted by irradiation with light of wavelength 424 mμ. (the principal absorption band of I) at −144°C. into a compound (II) having its principal absorption band at 454 mμ. II reverts spontaneously to I at high temperatures with an activation energy of 5·7 kcal. I and II show fluorescence bands at 440 and 470 mμ. respectively. C.O.C.

Structure and Light Absorption of the Reversibly Polymeric Forms of *pseudoiso*Cyanine
H. Zimmermann and G. Scheibe
Z. Elektrochem., **60**, 566–569 (1956): *Chem. Abs.*, **50**, 16383 (25 Nov. 1956)

Study of the visible absorption spectra of γ-*iso*cyanine chloride—



in water at 20°C. and in ethyl alcohol at −183°C. and their determination as a function of concentration (10^{-5}–3×10^{-2}M.) show that not more than 3 mol. are required to produce the "polymeric absorption band". "Reversible polymerisates" containing > 3 mol. have the centre of the band displaced to longer wavelengths by as much as 20A. C.O.C.

Phosphorescence and Fluorescence of some Aromatic Nitro-amines
R. Foster, D. Ll. Hammick, G. M. Hood, and A. C. E. Sanders
J.C.S., 4865–4868 (Dec. 1956)

The phosphorescence and fluorescence of soln. of twenty-three methyl- and ethyl-subst. *p*-nitroanilines in a mixture of methyl and ethyl alcohols have been measured in the region 400–800 mμ. at ca. −180°C. The effects of methyl and ethyl groups are noted and discussed, particularly in the light of their possible steric effects. H.H.H.

Dye–Gelatin Phosphors. III— Saturation Characteristics of Phosphorescence
R. Iwaki
J. Chem. Soc. Japan, Pure Chem. Sectn., **77**, 801–804 (1956): *Chem. Abs.*, **50**, 16405 (25 Nov. 1956)

Study of the saturation characteristics of the phosphorescence of trypaflavin–gelatin phosphor shows that the degree, *S*, of phosphorescence saturation (i.e. ratio of number of excited molecules to the total number of molecules) could be expressed by—

$$S = 1 - \frac{1}{\left(1 + \frac{AI_0}{B}\right)^{\frac{1}{2}}}$$

(*A* = probability of excitation of dye molecules; *B* = transition probability of the reverse process; I_0 = intensity of the exciting light). This equation is based on a unimolecular reaction mechanism and takes into account the anisotropy of dye molecules. C.O.C.

Cyclisation of Nitriles. II— Formation of Anthracene Derivatives
C. K. Bradsher and D. J. Beavers
J. Org. Chem., **21**, 1067–1068 (Oct. 1956)

Cyclisation of *o*-benzylbenzonitriles with cold H_2SO_4 yields 9-aminoanthracenes, which readily undergo air-oxidation. H.E.N.

Recent Developments in Anthraquinonoid Acid and Direct Dyes
J. Günthard
Textil-Rund., **11**, 639–644 (Nov. 1956)

The constitutions of the principal anthraquinone acid dyes existing around 1925, which are described, have since served chemists as starting points from which to construct dye molecules for the following purposes— (1) extension of the range of colours from blue to violet, red, and yellow on the one hand and from blue to green and grey on the other; (2) bulk requirements for fast-to-milling dyes; and (3) development of levelling dyes. The ways in which these aims have been achieved are described with structural formulae and constitutions. In conclusion, it is shown how the anthraquinone acid dyes have been modified to give substantive blues and greens for cotton, a new and promising field of research. S.R.C.

Infrared Absorption Spectra of the Carbonyl Group in Methoxyanthraquinones, and in the Hydrochlorides of 1-Methoxyanthraquinones
L. A. Wiles and L. C. Thomas
J.C.S., 4811–4814 (Dec. 1956)

The carbonyl stretching frequency in anthraquinones is lowered by a methoxyl group *para* to a carbonyl group, and the hydrochlorides of 1-methoxyanthraquinones are found not to have a chelate ring structure as hitherto supposed. The polyhydrochlorides show an increase in the carbonyl frequency, and a possible explanation of this unexpected observation is that the additional HCl mol. are associated with the π-electrons of the aromatic nuclei, thereby reducing the polar nature of the carbonyl group. H.H.H.

[Some 4-Substituted 7-Methylisatins]
H. Mix and H. W. Krause
Chem. Ber., **89**, 2630–2636 (Nov. 1956)

Naphthaquinone Pigments in the Tropical Sea Urchin *Diadema antillarum* (Philippi)
N. Millott
Nature, **178**, 1185 (24 Nov. 1956)

Synthesis of the Uroporphyrins II and IV
S. R. MacDonald and K. H. Michl
Canadian J. Chem., **34**, 1768–1781 (Dec. 1956)

Visual Pigments of Deep-sea Fish
E. J. Denton and F. J. Warren
Nature, **178**, 1059 (10 Nov. 1956)

Flavonoids of Citrus. I— Isolation of Diosmin from Lemons (*Citrus limon*)
R. H. Horowitz
J. Org. Chem., **21**, 1184–1185 (Oct. 1956)

Pearl Essence
M. Perpar and J. Beraus
Kemija u Industriji (Zagreb), **5**, 37–39 (1956)

For preparation of pearl essence (*C.I.* Natural White 1), guanine was extracted from the scales of *Squatius cephalus*, *Alburnus scorarizoides*, and *Trutta ochridana*. The scales were treated with water containing some ammonia, and filtered through cloth, and the guanine crystals were separated from the filtrate by centrifuging. They were treated with pepsin for 7 days in acid solution and then purified in organic solvents, e.g. amyl acetate and ether. The scales of *T. ochridana* gave the best product. C.O.C.

Zolon Red and the Zolon Red–Silver Complex

G. F. Svatos and J. Goldenson

J. Org. Chem., **21**, 1171–1173 (Oct. 1956)

Fastness to Light of Lead Stannate

E. Denninger

Maltechnik, (2), **38** (1955): *Chem. Abs.*, **50**, 17470 (25 Nov. 1956)

Lead stannate was in use as an artist's pigment from the 15th to the 17th centuries. It is prepared by heating red lead (3 parts) with tin oxide (1 part). At 600°c. an orange pigment is produced, at 725°c. a canary yellow, and at 800°c. a lemon yellow. Each of these three pigments together with Naples Yellow (Pb antimoniate) as control were painted out with Glutolin on a chalk ground and exposed to direct sunlight under glass for 22 weeks. No fading or colour change was observed, nor after 100 hr. exposure to ultraviolet radiation. C.O.C.

Effect of Inorganic Additions on the Colour of the "Red Iron Oxide" produced by Thermal Decomposition of Ferrous Sulphate— III

L. N. Uspenskaya and A. Kh. Girenko

J. Appl. Chem. U.S.S.R., **29**, 1142–1147 (Aug. 1956)

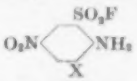
Metallic salts present in any quantity in $FeSO_4$ produce deterioration of the colour of the "red iron oxide" pigment produced from it. The greatest deterioration in the shade was found to be due to those cations whose radii differ greatly from the radius of Fe^{2+}, which is 0·67 μ. The presence of oxygen in the initial stages of dissociation of $FeSO_4$ causes increase in brightness and decrease in tone of the resulting pigment. The presence of sulphur causes some deterioration of the colour. T.Z.W.

PATENTS

Aminonitrobenzenesulphonyl Fluorides— Intermediates for Disperse Dyes

S *BP* 764,309

Aminonitrobenzenesulphonyl fluorides—



(X = H, Hal, lower Alk, lower OAlk, CF_3, or NO_2) are prepared by treating the corresponding sulphonyl chlorides with salts of HF in aqueous media. Thus 2-amino-5-nitrobenzenesulphonyl chloride is boiled with aq. NaF. E.S.

Monoazo Disperse Dyes having a Sulphonyl Fluoride Group

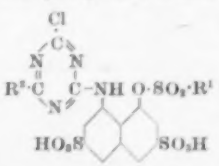
S *BP* 764,308

The 2-amino-5-nitrobenzenesulphonyl fluorides of *BP* 764,309 (*above*) are diazotised and coupled with suitable tertiary amines of the benzene series to give monoazo disperse dyes, which include violets and blues of good light fastness. Thus the monoazo compound 2-amino-3-chloro-5-nitrobenzenesulphonyl fluoride→*NN*-bishydroxyethyl-*p*-cresidine dyes cellulose acetate dischargeable violet. E.S.

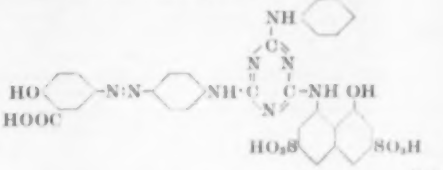
N-Triazyl Derivatives of H Acid— Intermediates for Azo Dyes

General Aniline *USP* 2,728,761

The condensation products of 1 mol. of cyanuric chloride (which may first be condensed with e.g. a primary amine) and 1 mol. of an *O*-sulphonyl ester of H acid



(R^1 = Alk, aryl, or aralkyl; R^2 = Cl or subst. amino radical) separate readily in a good state of purity. The remaining Cl atom may be replaced by reacting with an aminoazo compound, and the $SO_2{\cdot}R^1$ group may then be hydrolysed, giving azo dyes. Thus cyanuric chloride is condensed with an aqueous soln. of *O*-benzenesulphonyl-H acid cold by addition of aq. Na_2CO_3. At 40°c. condensation is effected with 1 mol. of 5-*p*-aminophenylazosalicylic acid, and the third Cl atom is reacted by treatment with aniline at 90–100°c. Hydrolysis of the benzenesulphonyl residue by heating at 70°c. with aq. NaOH then gives—

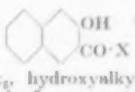


E.S.

Amides of 3:2-Hydroxynaphthoic Acid— Coupling Components for Azoic Dyes on Polyester Fibres

BASF *BP* 766,298

3:2-Hydroxynaphthoamide (I) and its *N*-alkyl, etc. derivatives—



(X = NHAlk, $NAlk_2$, hydroxyalkylamino, a hydrazine group, or the radical of a saturated *N*-containing ring compound), which may also carry NO_2, Hal, Alk or hydroxyalkyl groups in the naphthalene ring, may be applied to polyester fibres along with a suitable diazotisable amine, and an azoic dye produced by subsequent treatment of the fibre in aq. nitrous acid at 80–100°c. Thus I and *o*-aminoazotoluene give a bordeaux red on polyethylene glycol terephthalate. E.S.

Red Calcium Lake of Monoazo Dye

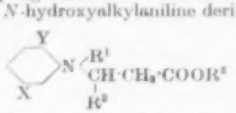
DuP *USP* 2,727,888

The calcium lake of the monoazo compound 4-amino-2-chlorotoluene-5-sulphonic acid→3-hydroxy-2-naphthoic acid, prepared by carrying out the coupling in presence of $Ca(OH)_2$, is converted into a desirable bluish tone of red by heating at 70–100°c. in presence of aq. alkali-metal hydroxides, alkali-metal carbonates, or $Ca(OH)_2$. E.S.

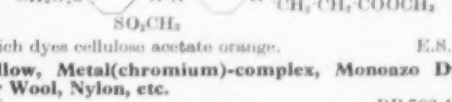
Orange and Red Monoazo Disperse Dyes

Eastman Kodak Co. *USP* 2,723,264

Orange and red monoazo dyes for cellulose acetate are made by coupling diazotised 2:4-bismethylsulphonylaniline (I) with *N*-hydroxyalkylaniline derivatives—



(R^1 = hydroxyalkyl of 2 or 3C; $R^2 = CH_3$, C_2H_5, or C_2H_4OH; R^3 = H or CH_3; X = H, Cl, Br, or CH_3; Y = H, or, when X = Cl or Br, Y also = OCH_3, OC_2H_5, or OC_2H_4OH). Thus I is diazotised in nitrosylsulphuric acid in presence of acetic acid, and the diazonium soln. diluted with crushed ice before adding slowly to a soln. in aq. HCl of *N*-β-hydroxyethyl-*N*-β-carbomethoxyethylaniline, to give—



which dyes cellulose acetate orange. E.S.

Yellow, Metal(chromium)-complex, Monoazo Dye for Wool, Nylon, etc.

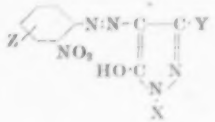
ICI *BP* 762,417

The complex containing 1 atom of Cr to 2 mol. of the monoazo dye (I) anthranilic acid→3-methyl-1-*o*-toluyl-5-pyrazolone dyes wool, nylon, etc. yellow from a neutral or weakly acid bath. Thus the Na salt of I is added to aq. ethylene glycol, chromium triacetate is added, and the mixture refluxed for 16 hr. E.S.

Yellow Spirit- and Lacquer-soluble Monoazo Pyrazolone Dyes

FH *BP* 763,555

The salts with organic bases of monoazo pyrazolone compounds—

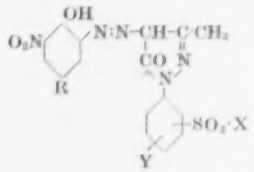


(X = H or Ar; Y = Alk, phenyl, COOH, $CONH_2$, or COOAlk; Z = H, Hal, Alk, CF_3, or SO_2H), having at least one COOH or SO_2H group, are spirit- and lacquer-soluble yellows, faster to light than the similar dyes of *BP* 277,371 and (293,067 (*J.S.D.C.*, **45**, 158 (1929); **48**, 340 (1932)) which have no NO_2 group *ortho* to the azo link. Thus diazotised *o*-nitroaniline is coupled with 3-methyl-1-*o*-sulphophenyl-5-pyrazolone. The monoazo compound so formed is dissolved in water and a soln. of di*cyclo*hexylamine sulphate added, whereupon the lacquer-soluble salt separates. E.S.

Metal(Chromium)-complex Monoazo Pyrazolone Dyes for Wool, Nylon, etc.

Gy *BP* 762,905

The solubility and degree of exhaustion of the complexes from 1 atom of Cr and 2 mol. of monoazo compounds—

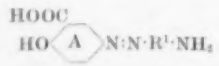


(R = Alk of 1–8 C; X = CH_3, NH_2, $NHCH_3$, or $NH{\cdot}C_2H_4OH$; Y = H, CH_3, or Cl) are largely influenced by the size of the group R. Thus if R is small, e.g. CH_3, the complex has poor exhaustion from a neutral bath, but good solubility, whereas if R has 4–8 C the complex has little solubility but good exhaustion from a neutral bath. Products combining satisfactory solubility with good exhaustion are obtained by combining two such monoazo compounds, in one of which R has 4–8 C, and in the other only 1 C, with 1 atom of Cr. Thus 1 mol. of the monoazo compound from diazotised 2-amino-6-nitro-4-*tert.*-amylphenol and 3-methyl-1-(4′-methyl-3′-sulphamylphenyl)-5-pyrazolone and 1 mol. of the monoazo compound from the same coupling component and diazotised 2-amino-4-methyl-6-nitrophenol are mixed together in aq. ammonia and stirred at 95–100°C. with ammonium disalicylato chromate. The chromium complex so formed dyes wool bluish red from a neutral or weakly acid bath. E.S.

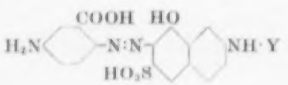
Copperable Disazo Direct Dyes

Ciba *BP* 760,347

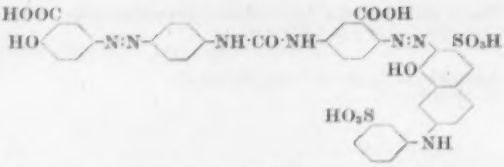
An aminomonoazo compound—



(R^1 = benzene or naphthalene radical in which the azo and NH_2 groups are *para* to one another; A may contain other substituents) is condensed with another aminomonoazo compound—



(Y = aryl of benzene series containing a group imparting solubility in water) by means of phosgene or cyanuric chloride. Or the urea derivative may be made by converting one of the aminomononazo compounds into the phenylurethan by means of phenyl chloroformate followed by condensation with the second aminoazo compound. The disazo compounds so formed are direct dyes which may be coppered in substance or on the fibre. When coppering in substance only 1 atom of Cu per mol. of dye is preferably used so that complex formation takes place at the *o*-carboxy-*o′*-hydroxyazo group but not at the salicylic acid residue. Thus the acetyl group is removed from the monoazo dye 5-acetylaminoanthranilic acid → *N*-*m*-sulphophenyl-γ acid by alkaline hydrolysis, and the product dissolved in aq. Na_2CO_3 together with 4-amino-4′-hydroxyazobenzene-3′-carboxylic acid. Phosgene is then passed in at 35–40°C. to give the disazo compound—



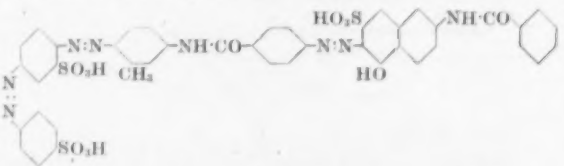
Heating with aq. $CuSO_4$ gives the complex containing 1 atom Cu to 1 mol. of disazo compound; it dyes cotton brown. E.S.

Orange, Red, and Brown Trisazo Direct Dyes

S *BP* 762,844

Aminobenzoylaminodisazo compounds—

(R^1 and R^2 = radicals of benzene or naphthalene series free from OH, NO_2, and NH_2 groups and together contain two SO_3H groups, R^1 containing at least one SO_3H; one X = NH_2, the other X = H, Hal, Alk, or OAlk; the two azo groups are in the *para* position) are diazotised and coupled with *N*-acyl-J acid or *N*-acyl-γ-acid, to give dischargeable direct oranges, reds, and browns. If the acyl group present in the end component contains an NH_2 group, the dye formed may be diazotised on the fibre and coupled with e.g. β-naphthol. Thus 4-aminoazobenzene-3:4′-disulphonic acid is diazotised and coupled with *m*-toluidine, and the product condensed with *p*-nitrobenzoyl chloride. Reduction of the nitro group gives the aminobenzoylaminodisazo compound, which is diazotised and coupled with *N*-benzoyl-J acid to give the trisazo compound—

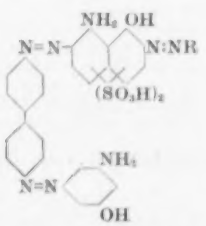


It dyes cotton dischargeable yellowish red. E.S.

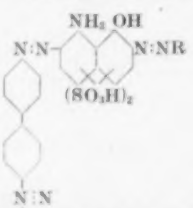
Deep Black Leather Dyes

CFM *BP* 765,087

Full neutral blacks are obtained on leather with dyes of formula—



produced by treating a diazodisazo dye of formula—



acid radical with 1-amino-3-hydroxybenzene.

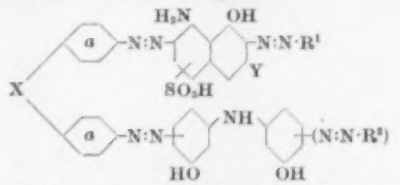
(R = naphthalene or benzene substituted by Alk, alkoxy, Ar, aryloxy, Hal, NO_2, acylamino, NH_2 or COOH but no SO_3H). Thus a trisazo dye is obtained by acidic coupling of 4:4′-tetrazodiphenyl with 1-amino-8-hydroxynaphthalene-3:6-disulphonic acid, combining in alkaline medium

with diazobenzene, and coupling the resulting diazo-disazo dye with 1-amino-3-hydroxybenzene. The product dyes chrome leather a full neutral black from a neutral or slightly acidic liquor. It is unaffected by hardness in the water. C.O.C.

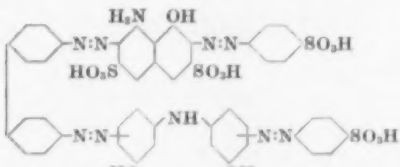
Trisazo and Tetrakisazo Dyes for Leather

FH *BP* 763,064

Trisazo and tetrakisazo compounds—



(R^1 and R^2 = aryl residues; X = direct link, or CH:CH, NH·CO·NH, CO, O, S, NH, or CONH; Y = H or SO_3H; the phenyl radicals *a* may carry substituents) dye leather deep violet, blue, green and black, and are resistant to acids, alkalis, and formaldehyde. Thus tetrazotised benzidine is coupled under acid conditions with 1 mol. of H acid. After neutralising, diazotised sulphanilic acid is added, and coupling completed by adding soda ash. A weakly alkaline soln. of 3:3′-dihydroxydiphenylamine is then added and coupling conducted at pH 10 by addition of 2N NaOH soln. Finally diazotised sulphanilic acid is coupled with the trisazo compound to give the tetrakisazo dye—

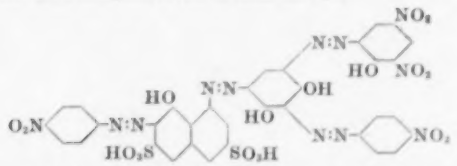


It has good solubility in water and dil. acids, and dyes chrome-tanned leather deep black. E.S.

Metal(Iron)-complex Tetrakisazo Brown for Leather

FBy *BP* 764,051

The iron complex of the tetrakisazo dye—

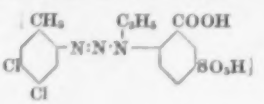


dyes bright, full browns on leathers of various tannages. It is made e.g. by coupling diazotised H acid with resorcinol, coupling the monoazo compound so formed with diazotised 2-amino-4:6-dinitrophenol, and coupling the disazo dye so formed with 2 mol. of diazotised *p*-nitroaniline. E.S.

Diazoamino Compounds Giving Azoic Dyes on Neutral Steaming

By *BP* 762,269

The diazoamino compounds obtained by combining diazotised alkyl (or alkoxy)-dihalogenoanilines with 2-alkylamino-5 (or 4)-sulphobenzoic acids readily regenerate the diazo compound on neutral steaming, but show good stability in the printing paste. They may be used for the production of azoic prints side by side with vat colours, without the decrease in strength often exhibited by azoic combinations under these conditions. Thus 1 mol. of diazotised 2-amino-4:5-dichlorotoluene is run slowly, together with aq. Na_2CO_3, into a weakly alkaline soln. of 2-ethylamino-5-sulphobenzoic acid to give—

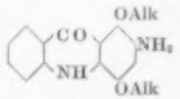


which is salted out in the form of its Na salt. This is mixed with 3-hydroxy-2-naphtho-*o*-phenetidide dissolved in aq. alcoholic NaOH, suitably thickened, and printed on to a cotton or other cellulose fibre. After drying, development for 5–10 minutes in neutral steam gives the scarlet azoic dye. E.S.

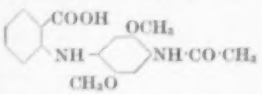
Greenish-blue Azoic Dyes from Aminoacridones

American Cyanamid Co. *USP* 2,725,375

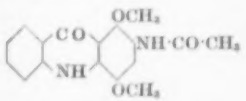
The aminoacridones of *USP* 2,694,713 (J.S.D.C., **71**, 680 (1955))



may be diazotised and coupled on the fibre with e.g. arylides of 3:2-hydroxynaphthoic acid to give greenish blues. Thus 4-amino-2:5-dimethoxyacetanilide is condensed with 2-bromobenzoic acid by refluxing in amyl alcohol with K_2CO_3, Cu, and I_2 to give—



Treatment with PCl_5 in benzene, followed by addition of $SnCl_4$, gives the acridone—

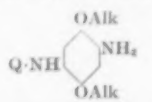


Hydrolysis with aq. alcoholic KOH removes the CO·CH_3 group giving the aminoacridone, which may be diazotised and coupled on cotton with 3-hydroxy-2-naphthoanilide to give a blue azoic dye. E.S.

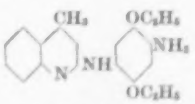
Greenish-blue Azoic Dyes from Quinolylamino Derivatives of Aniline

American Cyanamid Co. *USP* 2,725,376

The amines of *USP* 2,694,714 (J.S.D.C., **71**, 680 (1955))—



(Q = a radical of 2-quinoline and its homologues) may be diazotised and coupled on the fibre with arylides of 3-hydroxy-2-naphthoic acid to give greenish blues. Thus 4-amino-2:5-diethoxyacetanilide is condensed with 2-chlorolepidine by refluxing in aq. dioxane in presence of HCl. Hydrolysis with aq. alcoholic HCl then gives the amino compound—

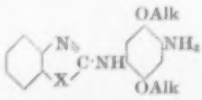


which may be diazotised and coupled on cotton with 3-hydroxy-2-naphthoanilide to give green blue. E.S.

Greenish-blue Azoic Dyes from Amino-oxazole and -thiazole Derivatives

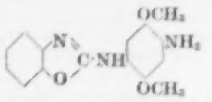
American Cyanamid Co. *USP* 2,725,377

The amines of *USP* 2,695,298—



(X = O or S) may be diazotised and coupled on the fibre with e.g. arylides of 3:2-hydroxynaphthoic acid to give greenish-blue azoic dyeings. Thus 4-amino-2:5-dimethoxyacetanilide is condensed with 2-chlorobenzoxazole by boiling in aq. dioxane in presence of HCl, and the produc-

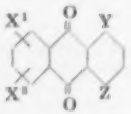
hydrolysed in aq. alcoholic HCl to give the amino compound—



which may be diazotised and coupled on cotton with 3-hydroxy-2-naphthoanilide to give a very greenish blue. E.S.

Alkylaminoanthraquinones
American Cyanamid Co. *USP* 2,727,045

Compounds of formula—



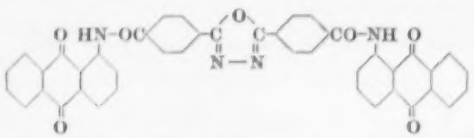
(X^1 and X^2 = same or different, H, Hal, OH, cyano or nitro; Y = amino, monoalkylamino or monohydroxyalkylamino; Z = OH, amino, monoalkylamino or monohydroxyalkylamino) are dyes for cellulose acetate, plastics, petrol and oils, and having high thermal stability, are used in coloured smokes and other pyrotechnics. They are rapidly obtained in good yield by condensing quinizarine in presence of a phenol with ammonia, an amine or mixtures thereof. C.O.C.

Yellow Pigment
Interchemical Corpn. *USP* 2,727,044

A bright reddish yellow pigment of excellent fastness to light, washing and dry cleaning is obtained by treating 1 mol. symmetrical *o*-phthalyldichloride with 2 mol. 1-aminoanthraquinone preferably at > 110°c. Both components should be pure. C.O.C.

Oxadiazoleacylaminoanthraquinones— Vat Dyes
Ciba *BP* 762,149

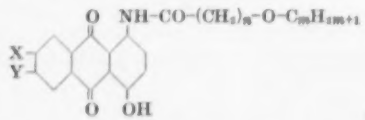
Yellow to red non-tendering vat dyes of formula—



and related ones in which the phenylene-oxadiazole feature is repeated and/or the anthraquinone residues are different and/or carry acylamino groups in the 5-position are made e.g. by condensing the acid chloride prepared by heating 2:5-bis[4′-carboxyphenyl-(1′)]-1:3:4-oxadiazole with $SOCl_2$ in trichlorobenzene containing pyridine, with 1-aminoanthraquinone at 180–190°c. in the same solvent. R.K.F.

Acylamino-anthraquinone Dyes for Polyester Fibres
S *BP* 764,030

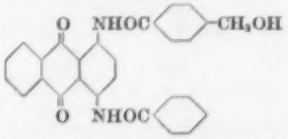
Orange and reddish-orange dyes for polyester fibres are produced by reacting 1-amino-4-hydroxyanthraquinone and certain derivatives thereof with alkoxy fatty acid derivatives. The dyes are of formula—



(X and Y = same or different, H, Fl, Cl or Br; n = 2–5; m = 1–8). Thus, to a dehydrated solution 1-amino-4-hydroxy-anthraquinone (12 parts) in chlorobenzene (300) there is added at 80°c. dry 2-*iso*-amyloxypropionic acid (9) and phosphorus trichloride (6). The mixture is stirred at 110°c. until a chromatographic test shows the starting material to have disappeared. The mixture is cooled, poured into water (400), neutralised with Na_2CO_3 and the solvent removed by steam distillation. The dye is filtered off, washed and dried. It dyes acetate rayon and Terylene orange, fast to light and gas-fume fading. E.T.

Acylaminoanthraquinone Vat Dyes containing the Hydroxymethylbenzoylamino Group
FBy *BP* 762,888

Vat dyes with outstanding vattability are produced by acylating aminoanthraquinones, aminoanthrimides and aminoanthrimide carbazoles with acetoxymethyl benzoyl chloride or *w*-chloromethylbenzoyl chloride followed by converting the acetoxymethyl group or the *w*-chloromethyl group to $-CH_2OH$. Thus, 1-amino-5-benzoylamino anthraquinone in nitrobenzene at 100°c. is treated with *p*-acetoxymethylbenzoyl chloride in nitrobenzene. The product formed by further heating for 3–4 hr. at 135–40°c. is filtered off and hydrolysed by treatment with 90–98% sulphuric acid at 4–10°c. to yield—



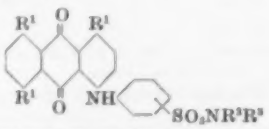
The rate of fixation of the vat dyes so made is greater than that of analogous products containing no hydroxymethyl group. E.T.

Acylaminoanthraquinones— Vat Dyes
Ciba *BP* 762,752

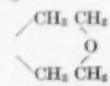
Yellow vat dyes are produced by condensing 1:5-diaminoanthraquinone (2 mol.) with an aromatic dicarboxylic acid dichloride (1 mol.) and further acylating with a monocarboxylic acid. Thus 1:5-diaminoanthraquinone is heated for 7–8 hr. at 150–160°c. with *iso*phthaloyl dichloride in trichlorobenzene containing pyridine. Na_2CO_3 is added, the pyridine distilled off and benzoyl chloride added at 190–200°c. for 30 min. R.K.F.

Blue to Bluish Green Anthraquinone Disperse Dyes of Good Fastness to Gas Fume Fading
General Aniline *USP* 2,727,903

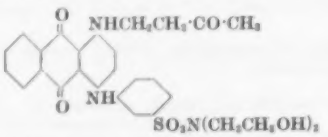
Dyes of formula—



(one R^1 = $NH{\cdot}CH_2CH_2{\cdot}CO{\cdot}CH_2{\cdot}X$, the other R^1s = H; X = H, OH, Alk or acyloxy; R^2 and R^3 = H, Alk, hydroxyalkyl, hydroxyalkoxyalkyl, hydroxypolyalkoxyalkyl, cyanoalkyl or together =



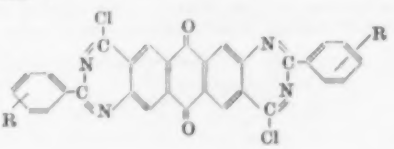
are blue to bluish-green disperse dyes of good fastness to light, washing and gas fume fading. Thus N^2-[4-aminoanthraquinonyl]$N'N'$-bis(2-hydroxyethyl)metanilamide, glacial acetic acid and methylvinylketone stirred together at 90°c. yield—



which dyes acetate rayon greenish blue. C.O.C.

Anthraquinone Vat Dyes
BASF *BP* 763,328

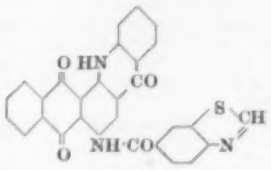
Yellow, greenish-yellow and green vat dyes are made by condensing 1 mol. of a bis-chloro-quinazoline compound of formula—



(R = H, Alk or Hal) with about 2 mol. of a vattable aminoanthraquinone compound, e.g. 1-aminoanthraquinone or 1-amino-5-benzoylaminoanthraquinone. The bis-chloro-quinazoline compounds are made by the method of *BP* 642,129 (J.S.D.C., **67**, 37 (1951)) and they are condensed with the aminoanthraquinone compound by heating at 100–230°C. in an inert solvent. Purification is effected by forming the sulphate of the dye. The dyes produced have excellent fastness properties, in particular high fastness to light. E.T.

Acylaminophthaloylacridones— Vat Dyes
CFM *BP* 761,687

A benzthiazole-6-carboxylic acid chloride substituted in the 2-position by H, Alk, Ar or heterocyclic radical is condensed with 6-amino-3′:4′-phthaloyl-acridone to give blue vat dyes. Thus 2-phenylbenzthiazole-6-carboxylic acid chloride is heated at 130–200°C. for 6 hr. with 6-amino-3′:4′-phthaloyl-acridone in *o*-dichlorobenzene to give the dye—



R.K.F.

Solvent-stable Copper Phthalocyanine Pigments
BASF *BP* 761,718

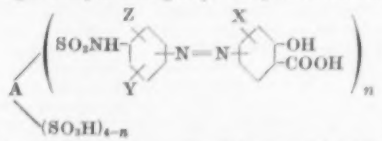
Copper phthalocyanine in the α-form is prepared in a form stable to the solvents used in lacquer making by dissolving the unsubstituted copper phthalocyanine in H_2SO_4 along with an amount of the corresponding chloro-, nitro-, or amino-derivative (substituted in the 4-positions), sufficient to give a mixture, on pouring into hot water, which contains 0·7–6% Cl, 1–7% NO_2 or 0·3–3% NH_2.
R.K.F.

Monotriazole Fluorescent Brightening Agents
Gy *BP* 763,696

When preparing 2-stilbyl-(naphtho-1′:2′:4:5)-1:2:3-triazole compounds (use of which as fluorescent brightening agents is described in *BP* 717,889 (J.S.D.C., **71**, 60 (1955))) handling of the toxic 2-aminonaphthalene is avoided by coupling diazotised 4-aminostilbene-2-sulphonic acids with a sulphonated derivative of 2-aminonaphthalene coupling in the 1-position which splits off or allows replacement of the sulphuric acid group or the radical bearing it in one of the steps of the triazole formation. C.O.C.

Water-soluble Mordant Azophthalocyanine Dyes
DH *BP* 763,343

Green to olive dyes suitable for textile use are made by condensing metalliferous phthalocyanine tetra-sulphochlorides with 1–4 mol. of an amino-azo compound containing a salicylic acid group. They have formula—

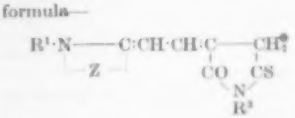


(A = subst. or unsubst. metalliferous phthalocyanine residue; *n* = 1–4; X = H, Hal, CH_3, OH, NO_2 or SO_3H; Y = H, Hal, Alk, alkoxy, NO_2, SO_3H or COOH; Z = H, Hal, Alk, or alkoxy).

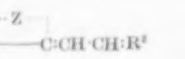
The $-SO_2NH-$ group is in *m*- or *p*-position to the azo group. The metalliferous phthalocyanine tetrasulphochloride is obtained either by starting from phthalimide in which there is an $-SO_2Cl$ or $-SO_3H$ group in the 3- or 4-position *or* by reacting a metalliferous phthalocyanine with chlorosulphonic acid. Condensation with the amino-azo compound is carried out in aqueous medium using an acid binding agent. The phthalocyanine tetrasulphochloride may react with 1–4 mol. of the azo compound and any $-SO_2Cl$ groups which do not so react are hydrolysed to $-SO_3H$ groups. Products containing greater numbers of sulphonic acid groups show greater substantivity whilst those with more salicylic acid groups have more pronounced mordant properties. E.T.

***mero*Cyanine Dyes**
FH *BP* 764,690

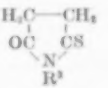
Dyes of formula—



are obtained by condensing a compound of formula—



with one of formula—



(Z = atoms to complete a 5- or 6-membered ring; R^1 = Alk; R^2 = O or $>N{\cdot}C_6H_5$; R^3 = CH_3, C_2H_5, allyl, *cyclo*hexyl, phenyl or benzyl). They can be used as photographic sensitisers. C.O.C.

Carbon Black
Columbian Carbon Co. *BP* 763,159

Carbon Black
Phillips Petroleum Co. *BP* 766,281

Modification of *BP* 679,818 (J.S.D.C., **68**, 528 (1952)) to enable production of an increased yield of pigments per unit of make hydrocarbon at any specific quality level. C.O.C.

Continuous Formation of Aqueous Slurries of Carbon Black
Columbian Carbon Co. *BP* 765,486

Panchromatically-sensitive Zinc Oxide
Radio Corpn. of America *USP* 2,727,807–8

A method of preparing zinc oxide that is photosensitive over the entire visible spectrum and particularly useful in electrostatic printing processes. C.O.C.

Aromatic Reactivity. I— Effects of Substituents on the Acid Cleavage of Phenyltrimethylsilanes (III p. 103)
Photoreaction of Reversible Fading of Dyes adsorbed on Microporous Glass (VIII p. 115)
Orange to Claret Dyeings on Acetate Rayon, Nylon or Polyurethane Fibre (VIII p. 116)
Black Electrostatic Dusting Powder (IX p. 116)
Pigments and Finishing Agents (XII p. 118)
Absorption Spectra of Leuco-base Dyes in Acid Solutions and in the Solid State. II— Leuco Bases of Thiazine and Oxazine Dyes in the Solid State (XIV p. 120)
Absorption Spectra of Sublimed Dye Layers (XIV p. 120)

V— PAINTS; ENAMELS; INKS

Microscopy of Heterogeneous Polymeric Systems
G. C. Claver and E. H. Merz
Off. Dig. Fed. Paint Varn. Prod. Clubs, **28**, 858–868 (Oct. 1956)

A review of work— some original, some already published— relating to the use of electron micrography and phase-contrast microscopy in the study of heterogeneous polymer systems. Multiphase polymer, polymer–polymer, pigment–polymer, and plasticiser–polymer systems are discussed. There are five photomicrographs and seven electron micrographs. J.W.D.

Influence of Solvents on Pigment Dispersion and Seeding
F. K. Daniel
Off. Dig. Fed. Paint Varn. Prod. Clubs, **28**, 837–857 (Oct. 1956)

"Seeding" or "pigment shock", which sometimes occurs when a paint vehicle is added to a mill base, is a complex phenomenon; it is not, however, as unpredictable as has been assumed. Strong aromatic solvents in the mill base often promote seeding, whilst aliphatic and slow solvents minimise it. The presence of the same solvent in both dispersion and vehicle produces more seeding than a two-solvent combination; the weaker solvent should be used in the dispersion. Seeding is a pigment agglomeration proc

caused by diffusion of solvent from the dispersion; diffusion is delayed by the use of weak solvents. Time of contact (during which diffusion can proceed undisturbed) may be shortened by internal agitation set up by differential surface tension between mill base and let-down vehicle. There are 17 photographs. J.W.D.

Blackening Effect of Hydrogen Sulphide on Exterior White House Paints
Pittsburgh Club Technical Committee
Off. Dig. Fed. Paint Varn. Prod. Clubs, **28**, 1089–1100 (Nov. 1956)

Blackening by the action of H_2S occurs only when Pb-containing pigments are present, it is independent of the nature of the Pb pigment used (it varies directly with Pb metal content), it does not demand a critical H_2S concn. in the atmosphere, and it does not occur unless the paint surface is wet with water— regardless of the R.H. of the atmosphere. A glossy and unweathered surface resists blackening for much longer than is the case with a paint having a high pigment vol. concn., or a weathered surface. Normal amounts of lead naphthenate drier in a Pb-pigment-free paint do not lead to appreciable discoloration. J.W.D.

Development of Emulsion Gloss Paints
A. C. Fletcher and J. E. O. Mayne
J. Oil & Col. Chem. Assocn., **39**, 923–933 (Dec. 1956)

Polyvinyl acetate (PVA) emulsions are considered as against oleoresinous and maleinised-oil vehicles. Photomicrographs, of which there are nine, show that the aq. phase has an important bearing upon PVA film characteristics. Titania pigments give max. gloss and coverage if they are dispersed in a plasticiser (or an emulsion of one) instead of in an aq. soln. of protective colloids. Polyacrylates or polymethacrylates are recommended as thickeners (where these are needed), and starch others to promote levelling. Gloss emulsion paints based upon PVA have several advantages over conventional gloss finishes— shorter drying time, freedom from odour, better alkali-resistance, and suitability for bright colours; their gloss is, however, somewhat inferior, and film hardness and application properties also require improvement. J.W.D.

Flooding and Floating Observations of Titanium Dioxide–Iron Blue Enamels
L. R. LeBrao, E. G. Bobalek, W. Von Fischer, and A. S. Powell
Off. Dig. Fed. Paint Varn. Prod. Clubs, **27**, 607–626 (1955): *Chem. Abs.*, **50**, 14470 (25 Nov. 1956)

Flooding and floating in paint films have been studied by spectrophotometry, electron microscopy, and sedimentation equilibria. An important variable is film thickness. There is preferential pigment migration both in liquid paint and in the dried film. Flocculates tend to settle much more slowly than smaller primary particles in a drying paint film. In thin enamel films the pigments distributed homogeneously, probably because there is quicker drying and so less opportunity for migration. In thicker films the iron blue migrates preferentially to the air interface and the TiO_2 to the film–substrate interface. C.O.C.

Wood Finishes and Wood Finishing
A. E. Lain
J. Oil & Col. Chem. Assocn., **39**, 670–694 (Sept. 1956)

Wood stains and staining are broadly discussed, after which the principles underlying the formulation of cellulose nitrate lacquers are treated in detail. Synthetic finishes are also considered, and their relative advantages and disadvantages as compared with cellulose nitrate finishes are examined in detail. Typical formulations for synthetic finishes based on butylated urea, epoxide resins, polyesters, and polyurethans are given, together with practical details of their application and performance. The assessment of film properties, such as bloom-resistance, rate of hardening, and durability, is discussed. J.W.D.

PATENTS

Dispersions of Powders
Dehydag Deutsche Hydrierwerke *BP* 763,367

Powders, e.g. pigments, are rendered more readily wettable or dispersible in liquid, highly viscous or plastic media, if they are mixed with the medium in presence of a salt of an acid phosphoric acid ester of an alcohol of > 6 C. Preferably the powder is impregnated with the salt before it is mixed with the medium. C.O.C.

Coloured Titaniferous Frits for Vitreous Enamels
National Lead Co. *BP* 764,728

A colour stain for use in vitreous enamels is obtained by calcining a mixture of a compound of a chromophoric metal and TiO_2. Enamels coloured with this stain may be fired and refired without alteration in colour. C.O.C.

Colouring Composition for Duplicating Inks
DuP *USP* 2,727,825

A mixture of several basic dyes, each in the form of its salt with a naphthalene polysulphonic acid, e.g. 1-naphthylamine-3:8-di(sodium sulphonate), which may also bear OH or NH_2 groups is used to colour duplicating inks which are to be non-smudging and non-staining. The salts are prepared by mixing aqueous solutions of the dye and the naphthalenepolysulphonic acid. The dyes used are chosen from C.I. Basic Violet 1, 3, 10, 13 and 14; C.I. Basic Blue 7 and 26; C.I. Basic Red 1 and 2; and C.I. Basic Green 1. C.O.C.

Emulsion Paints
Esso Research & Engineering Co. *BP* 765,408

Use of a synthetic hydrocarbon polymer drying oil, e.g. a copolymer of butadiene-1:3 with styrene, in an emulsion paint results in coatings which have remarkable resistance to washing and scrubbing and which do not materially change in colour. C.O.C.

Latex Coating Compositions containing Zinc Phosphate-modified Zinc Oxide
Dow Chemical Co. *USP* 2,727,012

Compositions, yielding coatings resistant to weathering, comprise an aqueous latex of a film-forming polymer, e.g. a copolymer of an aliphatic conjugated diolefin and a monovinyl aromatic hydrocarbon, mixed with a pigment composition containing 10–50% by weight of a phosphated zinc oxide pigment, are stabilised by presence of < 2, preferably 2·6–6·6, parts by weight per 100 parts pigment, of a β-oxyalkylamine oleate, e.g. monoethanolamineoleate. C.O.C.

Vitreous Enamels coloured with Ultramarine
General Electric Co. *USP* 2,726,965

Ultramarine and TiO_2 are ball milled with a frit made up of SiO_2 (8·5 parts by wt.), feldspar (17), TiO_2 (3), ZnO (29·5), CaF_2 (6), KNO_3 (6), H_3BO_3 (40), As_2O_3 (3·5), $BaCO_3$ (5), K_2CO_3 (5) and Na_2CO_3 (4) in presence of a suspension vehicle, e.g. alcohol. A satisfactory light-diffusing blue enamel highly transmissive of infrared radiation is obtained by milling 200 g. of the above frit with 40 g. ultramarine, 2–5 g. TiO_2 and 175 c.c. alcohol at 60 r.p.m. for 24 hr. The product can be diluted for application to electric lamp bulbs, the coated bulbs being baked for 3 min. at 720°C. after being dried. C.O.C.

VI— FIBRES; YARNS; FABRICS

Scorching Characteristics of Fabrics
AATCC Washington Section
Amer. Dyestuff Rep., **45**, P 845–P 856 (19 Nov. 1956)

The scorching characteristics of secondary cellulose acetate, cellulose triacetate, cotton, partially acetylated cotton, partially cyanoethylated cotton, Terylene, linen, nylon, Orlon, viscose rayon (filament and staple), cotton–rayon, silk, and wool were investigated, and the experimental technique is described. Measurement of reflectance and yellowing is necessary to describe scorching completely. Scorching is a function of time and temperature. Fabrics scorch faster in contact with heated metal than in air at the same temperature. Scorching is more dependent on chemical nature and finishing treatment applied to the fibre than on its physical nature. Tendency to scorch increases in the following order: acetylated cellulose fibres, native cellulose and polyester fibres, regenerated cellulose fibres, and fibres containing nitrogen. Low concentrations of weak alkalis increase scorching of cellulosic fabrics. Partial acetylation of cotton significantly increases its resistance to scorching. P.T.S.

Water-absorption Characteristics of Monofilament Cellulose
P. Denton
J. Textile Inst., **47**, T 570–T 585 (Nov. 1956)

Absorption is attributed to three processes— (i) diffusion from air stream to surface of filament, (ii) diffusion from surface to centre of filament through immediately accessible regions, and (iii) transport of water into regions made

accessible by interaction between them and water already absorbed in (ii). Change in diameter of section of filament and increase in weight are measured as water is absorbed. Curves of both quantities against time show discontinuity as (ii) is followed by (iii). Discontinuity coincides with diffraction bands reaching the centre of the filament from the outside. An approximate equation is derived for the process. Filaments 90–100 μ in diameter are used: in textile filaments of smaller diameter the importance of (iii) increases. Apparatus is described for measuring changes in cross-sectional area and regain. P.T.S.

Effect of Humidity on the Tensile Behaviour of Grey, Scoured, Mercerised, and Chemically Modified Cotton
R. Meredith

J. Textile Inst., **47**, T 499–T 510 (Oct. 1956)

Strength and breaking extension of grey, folded cotton yarn increase with R.H. Scoured and mercerised yarns give max. at 85% R.H. Yarns are treated with sodium hypochlorite, NaOH, or HCl, or are heated in air to 157°C. The max. moves to lower R.H. as tendering increases. For most highly tendered materials (as for viscose rayon) strength decreases with increase in R.H. The strength–humidity relationship is determined by average chain length of molecules as shown by viscosity in cuprammonium hydroxide. Parallel results are obtained with singles yarn, tyre cord, and fabric. In the range 45–85% R.H. the strength of grey cotton materials increases by 3–4% and the breaking extension by 6–7% for each 10% increase in R.H. P.T.S.

Chromatographic Study of Jute α-Cellulose
W. G. Macmillan, A. B. Sen Gupta, and A. S. Dutt

Nature, **178**, 1346–1347 (15 Dec. 1956)

Hydrolysis of jute α-cellulose by N. sulphuric acid, followed by neutralisation of the extract with barium carbonate and paper chromatography, indicated the presence of uronic acid, galactose, mannose, rhamnose, glucose, xylose, and arabinose. W.R.M.

Use of Radioisotopes in the Study of Wool Growth and Fibre Composition
M. L. Ryder

Nature, **178**, 1409–1410 (22 Dec. 1956)

Activity was detected in the vibrissal follicles of a mouse killed 2 min. after injection of cystine labelled with ^{35}S. Activity in the prekeratinisation zone was not detected until 2 hr. after injection. This suggests that compounds containing sulphur enter the follicle above the bulb. Results with lamb skin suggest that sulphur may enter the follicle in a compound containing thiol groups. It is also found that there is no significant difference between the autoradiographic grain density counts for the orthocortex and the paracortex, suggesting that these contain similar amounts of cystine. W.R.M.

Wool Fibre of Sheep infected with Flukes. I—Chemical Observations
I. Shimizu and S. Kobayashi

J. Soc. Textile Cellulose Ind. Japan, **12**, 727–731 (Oct. 1956)

Comparison of Australian merino wool with 13 kinds of Japanese wool shows that— (1) the content of kempy wool from the afflicted sheep is higher than in the merino; (2) the grease content is lower than in the merino; (3) the total nitrogen and diamino-acid nitrogen contents are lower, but amide nitrogen and monoamino-acid nitrogen contents are greater than in the merino; the total sulphur content is somewhat lower, and the tyrosine content lies between 3·3 and 6·4%; and (4) the affinity of acid dyes for Japanese wool is smaller, and that of basic dyes varies in some cases; acid-combining capacity is related to the affinity of basic dyes on Japanese wool. AUTHORS

Absorption of Alkali by Wool Keratin from Aqueous Solutions of Sodium Carbonate and Bicarbonate
R. P. Harker

J.S.D.C., **73**, 17–23 (Jan. 1957)

Sorption of alkali by wool from aqueous solutions of sodium carbonate and bicarbonate has been examined by measurement of hydrogen-ion concentrations. The effects of concentration of alkaline salt, time of immersion, liquor ratio, and temperature of equilibration have been studied in relation to subsequent measurement of the pH of an aqueous extract of a treated sample. A qualitative interpretation of results is given, and a quantitative treatment employing the Donnan theory of membrane equilibria has been attempted. AUTHOR

Use of Irradiated Polymers as Initiators of Polymerisations
J. C. Bevington and D. E. Eaves

Nature, **178**, 1112–1113 (17 Nov. 1956)

Polystyrene, polyacrylonitrile, Terylene, and nylon were irradiated with γ-rays, and the irradiated polymers exposed to the vapour of ^{14}C-acrylonitrile. Polyacrylonitrile were formed. It is believed that free radicals at, or very near, the surface of the irradiated polymer act as centres for the polymerisation of the ^{14}C-acrylonitrile. W.R.M.

Chemical Constitution and Textile Properties of Synthetic Macropolymers
G. Champetier

Teintex, **21**, 955–973 (Dec. 1956)

An attempt is made to relate the textile properties of fibres with their constitution and macromolecular structure. Crystallinity, cross-linking, D.P., polar groups, chain rigidity, copolymerisation, and copolycondensation are considered. S.R.C.

Isotactic Polymers
J. W. S. Hearle

Research, **9**, 461–465 (Nov. 1956)

Isotactic polymers are those possessing a spiral arrangement of side-groups. Their production is outlined, and some properties of fibres from isotactic polymers are considered. W.R.M.

Spinning Man-made Fibres from Organic Synthetic Polymers
H. Perner

Faserforsch. und Textiltech., **7**, 515–525 (Nov. 1956)

A review of the present state of development. W.R.M.

Equilibrium in the System: Water–6-Hexanolactam (ε-Caprolactam)
A. B. Meggy

J.C.S., 4876–4885 (Dec. 1956)

Equations are calculated for the activity coefficients of water and polymer in the system water–6-hexanolactam, and a composition–temp. diagram is constructed showing lines of equal D.P., of equal partial pressure of water, and of equal lactam content, from 200° to 300°C. H.H.H.

Determination of the Individual Cyclic Oligomers in Equilibrium Systems from 6-Hexanolactam (ε-Caprolactam) and Water
D. Heikens

Rec. Trav. chim., **75**, 1199–1204 (Sept.–Oct. 1956)

The contents of cyclic dimer, trimer, and tetramer in equilibrium polymerisates of ε-caprolactam and water have been determined by fractional sublimation (the apparatus is illustrated) of a methanol extract, freed from the cyclic monomer and linear oligomers. The proportions of the cyclic dimer, trimer, and tetramer in these products are 2 : 1 : 2 at 221·5°C. and 5 : 2 : 7 at 253·5°C. The results support the previously published theory of Hermans (*Nature*, **177**, 126 (1956)) that the high stability of amide groups in rings with an even number of repeating units may be due to the formation of internal hydrogen bonds between pairs of amide groups. The mean stability of the three amide groups in the trimer is lower than that of the amide groups in the dimer and tetramer. J.W.D.

Molecular Structure of High Polymers
XIV— Reaction of Polyamide Fibres with Bases
V. A. Myagkov and A. B. Pakshver

J. Appl. Chem. U.S.S.R., **29**, 1229–1235 (Aug. 1956)

Strong bases react with the carboxyl end-groups of the polyamide in stoichiometric proportions, forming salt linkages. On treatment of the polyamide with dilute solutions of basic dyes, only the cations of the dyes combine with the carboxyl groups of the fibre. The experimental data do not support the absorption theory of Gilbert and Rideal. The interaction of the carboxyl end-groups of the polyamide with bases proceeds by an ion-exchange mechanism, in which the carboxyl end-groups behave independently, regardless of the number of amino end-groups. The process of sorption of the base is followed by measurements of the concn. of the ions taking part in the exchange and the degree of dissociation of the reacting groups of the polyamide. The equilibrium constant of the

reaction increases with increase in size of the cation of the base.

XV—Absorption of Water Vapour by Capron [6-Nylon] Fibres
B. V. Petukhov and A. B. Pakshver
Ibid., 1236–1242

Oriented Capron fibres treated without tension with aqueous phenol show smaller absorption of water vapour at low R.H. Similar treatment under tension does not influence the absorption of water vapour. Thermal treatment decreases the absorption, the decrease being greater for heated fibres under tension or stretched. The process of strengthening of the intermolecular cross-linkages and condensation of the structure takes place very rapidly and is practically complete within 30 sec. The degree of absorption does not depend directly upon the orientation of the macromolecules, but also depends upon the number of intermolecular bonds between the macromolecules. The coefficient of diffusion for the oriented Capron fibres is lower than for the unoriented fibres and has a constant value in the low region of elasticity at 25% R.H. at 20°C. The coeff. of diffusion of water vapour depends on the number and the strength of the intermolecular cross-linkages and the density of the structure. The elasticity of Capron fibres can be considerably increased by heating the fibre under tension in superheated steam.

XVI—Reactivity of the Amide Groups of Polyamide Fibres
V. A. Myagkov and A. B. Pakshver
Ibid., 1242–1250

The amide groups of polyamides are able to react with acids, bases, and other hydroxy compounds in the undissociated form by means of hydrogen bonds according to the scheme—

$$-CO-NH- + R{\cdot}OH \rightleftharpoons -CO-NH- \quad (\uparrow HOR)$$

This process can be quantitatively represented by the equation—

$$K = \frac{\left[\begin{matrix}-NH-CO- \\ \uparrow \\ HOR\end{matrix}\right]}{[-NH{\cdot}CO-]\,[R{\cdot}OH]} = \frac{x}{(1-x)C_\infty}$$

HOR
↓
([–CO–NH–] = x = concn. of amide groups taking part in the reaction, in moles/residue; [–CO–NH–] = $1 - x$ = concn. of amide groups not taking part in the reaction; C_∞ = concn. of the reagent at equilibrium; and K = equilibrium constant). The equilibrium constants for the reactions of the amide groups with hydrochloric acid, piperidine, phenol, resorcinol, salicylic acid and tannins were calculated. The equilibrium constant for the reaction with phenol decreases with increase of stretch and characterises the stability of the bonds between the polyamide molecules. The equilibrium constant increases with decrease in the dissociation constant of the reagent, and can be taken as a measure of the "dissolving power" of the reagent. All the amide groups in polyamide fibres with a given degree of stretch are equivalent in reactions with substances which form hydrogen bonds. T.Z.W.

Preparation of Polyvinyl Chloride Fibre. XV—Effect on the Fibre Properties of the Degree of Polymerisation of the Spinning Solutions in the Wet Spinning of Polyvinyl Chloride–Tetrahydrofuran Solutions
T. Yoshioka
J. Soc. Textile Cellulose Ind. Japan, **12**, 700–703 (Oct. 1956)

Properties of Polyvinyl Alcohol Fibres
G. B. Carpenter and O. L. Wheeler
Canadian Textile J., **73**, 51–54 (19 Oct. 1956)
See J.S.D.C., **72**, 456 (Sept. 1956). A.H.

PATENTS

Insolubilising Regenerated Protein Fibres
ICI *BP* 763,501

A bundle of filaments, before or after being treated with a hardening agent, are treated with a compound which gives rise to a cyanate ion after which the treated hardened filaments are treated with HCHO, excess liquor removed and then heated while under tension until the filaments lose all tendency to shrink. Finally they are baked to effect insolubilisation. The products can be dyed with acid dyes in the normal manner, e.g. for 90 min. at 97°C. C.O.C.

Synthetic Resin Composition for producing Fibres having Good Affinity for Dyes
Polymer Industrie Chimiche *BP* 763,293

A mixture of a polymer and/or copolymer of vinyl chloride (> 70% vinyl chloride) with a polymer of a pyridine derivative containing at least one ethylenic double bond or a homologue thereof, can be dissolved to clear fibre-forming solutions. Fibres made from them can be dyed in the normal manner with acid dyes in absence of swelling agents. C.O.C.

Solutions of Acrylonitrile Polymers
Vereinigte Glanzstoff-Fabriken *BP* 765,549

A mixture of dimethylformamide and an acetal is an excellent solvent for producing solutions of acrylonitrile polymers from which filaments may be formed by either a wet or dry process. C.O.C.

Properties and Handling of Agilon Yarn (VIII p. 115)
Verel Acrylic Fibre (VIII p. 115)
Synthetic Linear Polymers. II—Viscometric Determination of the Molecular Weight of Polyamides in Aqueous Chloral Hydrate (XIV p. 120)

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Neutral Scouring of Raw Wool
R. A. Olney and B. A. Ryberg
Amer. Dyestuff Rep., **45**, 781–788 (22 Oct. 1956)

Overwhelming evidence is given for the superiority of scouring of raw wool with a non-ionic detergent and a builder in neutral solution over the conventional soap-and-soda method. Greater efficiency is possible, and a whiter, softer, and faster drying wool is produced. Results of research to discover the effect of detergent concn., type of builder (NaCl or Na_2SO_4), concn. of builder, and temperature are given. Higher temperatures can be used because of lower risk of damage in neutral solution. These results are confirmed in industrial tests. P.T.S.

Effect of Temperature and Period of Immersion in Backwashing
P. P. Townend and R. K. Pitcher
J. Textile Inst., **47**, T 611–T 612 (Nov. 1956)

The effect of raising the temperature of scouring above 125°F. is to cause a worse tear in combing and a shorter mean fibre length. Stoppages in the backwashing machine (which mean prolonging the immersion of the sliver in the liquor and also varying the period of drying of the sliver) have the same effects. P.T.S.

Alkali and Washing Treatments of Man-made Fibres
E. Dyhrenfurth
Textil-Rund., **11**, 573–587 (Oct.) and 656–668 (Nov. 1956)

The resistances of dynel, Vinyon N, Orlon, Terylene, Acrilan, and polyamide fibres to alkaline or neutral perborate decrease generally in that order, as measured by tensile strength. Terylene is only eroded by alkali but is chemically degraded by H_2SO_4. S.R.C.

Washing of Garments made of Synthetic Fibres
W. Breunecke
Textil Praxis, **11**, 1217–1220 (Dec. 1956)

The washing under domestic conditions of garments made from polyamide, polyacrylonitrile, polyester, and polyvinyl chloride fibres is discussed with reference to the chemical composition of the detergent used, the washing temp. employed, and their possible effects on the quality of the material. B.K.

PATENTS

Solvent Degreasing of Wool
Commonwealth Scientific & Industrial Research Organisation (Australia) *BP* 763,657

Multistage Washing
Boehme Fettchemie *BP* 765,811

Extremely good cleansing is obtained even of synthetic fibres material containing difficultly removable dirt by washing at < 60°C. with a liquid containing a cation-active

substance followed by at least one other washing with a liquid containing an anion-active substance. Thus nylon is treated first with an aqueous liquid containing 1·2 g./l. of sodium dodecylbenzenesulphonate and 0·5 g./l. of sodium tripolyphosphate, rinsed and then washed clear with 0·5 g./l. of hexadecyltrimethylammonium chloride and 1 g./l. acetic acid, the first liquid being at 20–60°c. and the second at 50°c. C.O.C.

Effect of Humidity on the Tensile Behaviour of Grey, Scoured, Mercerised, and Chemically Modified Cotton (VI p. 113)

Acrylic Fibres. XXIV— Chemical Properties (VIII below)

VIII— DYEING

Equilibrium Adsorption Values of Leuco Vat Dyes on Cotton

R. H. Peters and H. H. Sumner

J.S.D.C., **73**, 12–16 (Jan. 1957)

The equilibrium adsorption of a series of leuco vat dyes on cotton has been measured in various concentrations of sodium chloride and sodium hydroxide. From the results the thermodynamic affinities have been calculated. Deviations from theory are observed in so far as the affinities are not constant, but show a regular drift as the concentration of electrolyte is changed. This drift is more apparent when the added electrolyte is sodium chloride than when it is sodium hydroxide. Two explanations for these deviations from theory are proposed and discussed— either the theory is inadequate to account for the facts, or the leuco solutions of the dyes are not true solutions but are in fact aggregated. AUTHORS

Technology of Modern Oxidation Hair Dyes— III

G. S. Kass

Amer. Perfumer Aromat., **68**, (3), 47–50 (1956): *Chem. Abs.*, **50**, 17339 (25 Nov. 1956)

Many of the modern colouring preparations not only dye the hair but simultaneously bleach the natural pigments in it. Bleaching is effected with alkaline H_2O_2, which at the same time oxidises the oxidation dye bases present. Chlorates, persulphates, perborates, and Na dichromate are inferior to H_2O_2 for oxidising the dye bases in ease of use, completeness of oxidation, safety, and absence of undesirable byproducts. Detailed formulae for oxidation dyes for dark ash blonde, medium brown, and honey blonde are given. C.O.C.

High-temperature Dyeing in Forced-circulation Apparatus

B. Steverlynck

Teintex, **21**, 871–883 (Nov. 1956)

The method carrying the name of the author, with the Drijvers modification, is discussed at length, including its application to hydrophobic fibres and the use of dyes of poor solubility. S.R.C.

Effect of Resin Finishing on the Light Fastness of Vat Dyes

AATCC Piedmont Section

Amer. Dyestuff Rep., **45**, P 821–P 832 (5 Nov. 1956)

The effect of thermosetting resin finishes on the fastness of 30 vat dyes was investigated. Only 13 were unaffected. The effect was the same whether a cyclic ethyleneurea resin or a methylated melamine resin was used. The effect is not due to curing, nor catalyst, nor free formaldehyde. The $>N{\cdot}CH_2OH$ group in the resin appears to be responsible. The effect could not be correlated with dye structure. Washing after curing will reduce fading. The effect on mixtures of dyes cannot be predicted from their individual properties. P.T.S.

Properties and Handling of Agilon Yarn

N. C. Armitage

Canadian Textile J., **73**, 59–63 (19 Oct. 1956)

Some of the properties of bulked yarns are described, and recommended procedures are given for finishing and dyeing fabrics made from them. The principal uses to which these fibres have been put thus far are hosiery, sweaters, and carpet and rug manufacture. A.H.

Acrylic Fibres. XXIV— Chemical Properties

M. Katayama

J. Soc. Textile Cellulose Ind. Japan, **12**, 711–719 (Oct. 1956)

A study of the chemical properties of pure polyacrylonitrile and copolymer fibres has shown that— (1) HNO_2 has a bleaching action, (2) acrylamide copolymer fibres treated with HCl (> 20%) can be dyed with acid dyes, and (3) acrylic acid (CH_2:CH·COOH) copolymer treated with amine compounds shows good dyeability with acid dyes. AUTHOR

Verel Acrylic Fibre

R. T. Crawford and C. F. Earnhardt

Canadian Textile J., **73**, 56–60 (16 Nov. 1956)

An account of the properties of a new acrylic fibre manufactured by Eastman Chemical Products. Evaluation of fibre properties, and dyeing and finishing are discussed. There is also a table showing the action of a large range of chemicals on the fibre. A.H.

Photoreaction of Reversible Fading of Dyes adsorbed on Microporous Glass

A. V. Karyakin

Zhur. fiz. khim., **30**, 986–994 (1956): *Chem. Abs.*, **50**, 16410 (25 Nov. 1956)

Fading of dyes adsorbed on microporous glass, which is considered to be a form of silica gel, was caused by the reducing action of H from H_2O under the influence of light, and not by the destructive action of light in the dyes; this was proved by the colour being repeatedly restored by admission of O_2 after every reduction under the influence of light. The dyes used were— Methylene Blue, a thiazine dye (thiamine), and a vat dye (dibenzopyrenequinone). The pore surface of the glass was completely methylated by replacing all OH groups with $O{\cdot}CH_3$ groups. Adsorption measurements on regular glass samples, with OD replacing OH on the methylated pore surface, supplied data on the nature of the adsorption. Two types of adsorption centre were found— (*a*) one in which the OH group contains a high proportion of H^+, (*b*) one which may consist of stoichiometrically unsaturated Si atoms. At low R.H. adsorption is mainly in the second type of centres. At high R.H. the adsorption is in fact upon the OH groups. The force of interaction of the H_2O molecules with the OH groups is of the same order as in the liquid state, and much less than with the second class. The interaction of the OH groups and the adsorbed molecules results on the one hand in displacement of the absorption band of the adsorbed compound, and on the other in displacement and considerable widening of the absorption bands of the surface OH groups compared with the unexcited surface groups. The displacement of the bands increases with the rising proton-accepting (basic) properties of the adsorbed compounds. C.O.C.

Evaluation of Alkylated *p*-(*p*-Nitrophenylazo)anilines as Dyes for Petrol

J. W. Thompson and F. E. Johnson

Ind. Eng. Chem., **48**, 1869–1873 (Oct. 1956)

A series of alkylated *p*-(*p*-nitrophenylazo)anilines were prepared and evaluated as yellow and orange dyes for colouring petrol, in comparison with the standard dyes for this purpose (*NN*-dimethyl-*p*-phenylazoaniline (yellow) and 1-phenylazo-2-naphthol (orange)). *N*-Alkyl and ring-alkyl substitution had a pronounced effect on tinctorial strength and hue. Yellow dyes of tinctorial strength 32–450% of the standard yellow were obtained in the *N*-alkyl-*p*-(*p*-nitrophenylazo)aniline series as the alkyl increased from methyl to butyl. The *NN*-dialkyl-*p*-(*p*-nitrophenylazo)anilines above dimethyl were orange dyes. The best yellow and orange dyes among those studied were considered to be *N*-*sec*.-butyl-*p*-(*p*-nitrophenylazo)aniline and *NN*-dibutyl-*p*-(*p*-nitrophenylazo)aniline respectively. W.K.R.

PATENTS

Dyeing Polyethylene Terephthalate Fibre

DuP *USP* 2,727,803

The tarring often occurring when polyester fibres are dyed by the process described in *BP* 629,452 (*J.S.D.C.*, **66**, 161 (1950)) is avoided if the impregnation is carried out in two steps, viz. impregnation with the azo-diarylamine, rinsing to remove any loosely adhering diamine and any carrier used, followed by impregnation with an aqueous dispersion of 2:3-hydroxynaphthoic acid and final treatment with nitrous acid. C.O.C.

Colouring Polyacrylonitriles with Diarylmethane Dyes

Ciba *BP* 762,397

Salts of the dyes—

(R^1 = H or non-aromatic organic residue; R^2 and R^3 = Ar, one at least of which contains NH_2) are suitable for producing fast dyeings and prints on polyacrylonitrile fibres, e.g. Orlon 81. Thus bis(4-dimethylaminophenyl)-carbinol is mixed with chloroacetic acid and used to dye polyacrylonitrile staple for 1 hr. at 100°C. R.K.F.

Orange to Claret Dyeings on Cellulose Acetate, Nylon or Polyurethan Fibre
FH *BP* 765,740

The material is treated in an aqueous bath with an amine of formula—

(the benzene radical may be further substituted by Alk, alkoxy or Hal; X = CO, COO, SO_2, or SO_3; R = Alk, aralkyl, Ar or hydroaromatic) e.g. 1-amino-2-methoxybenzene-5-carboxylic acid-*n*-butyl ester and an alkali metal compound of a 2:3-hydroxynaphthoic acid arylide containing an alkoxy group in the arylide group, e.g. 1-(2′:3′-hydroxynaphthoylamino)-2-methyl-4-methoxybenzene, diazotising and then developing in a hot bath. The dyeings have a good fastness to cross dyeing. C.O.C.

Wool Fibre of Sheep infected with Flukes. I— Chemical Observations (VI p. 113)
Finishing of Man-made Fibres (X p. 117)

IX— PRINTING

Nylon Screens for Screen Printing
R. Künzl

Textil Praxis, **11**, 1115–1117 (Nov. 1956)

The properties of great strength, high elasticity and resistance to alkalis, lack of swelling, and water-repellency make nylon of value in the construction of screens for screen printing. Methods of preparing screens are described. B.K.

PATENTS

Light-sensitive Diazotype Layers containing Carboxamides
General Aniline *USP* 2,727,820

A layer for dry print material which gives higher developing speeds with no loss of maximum density. C.O.C.

Production of a Yellow Masking Image in Magenta Photographic Images
Agfa AG für Photofabrikation *BP* 765,280

When colour developing photographic layers containing magenta colour-forming couplers containing active methylene groups, any colour component not turned into a dye by the developer is reacted with pyrrole aldehyde, indole aldehyde, carbazole aldehyde or a functional derivative of one of these aldehydes to form a yellow styryl dye. C.O.C.

Dye Transfer Process particularly for Colour Photography
Kodak *BP* 763,797

A silver photographic image is bleached to a silver halide, and then dyed with a basic dye in presence of a water-soluble halide and finally washed free of excess dye and halide. The moist material is then pressed against a mordanted sheet in presence of a water-soluble silver salt thus transferring the dye imagewise from the image to the mordanted sheet by imbibition. C.O.C.

Photographic Filter Layers Irreversibly Dischargeable by Use of Photographic Bleaching Agents
General Aniline *USP* 2,727,821

Filter or antihalation layers produced from yellow Schiff's bases may be irreversibly discharged, e.g. with ferricyanide or acid bichromate solutions, if the Schiff's base used is obtained by condensing an *o*-hydroxyaldehyde of the benzene or naphthalene series with an *o*-phenylenediamine in which one amino group is primary and the other secondary, e.g. the azomethin from 2-hydroxynaphthaldehyde and 4-sulphomethylaminometanilic acid. C.O.C.

Black Electrostatic Dusting Powder
Radio Corpn. of America *USP* 2,727,826

A dye is precipitated onto a fine powder having good dielectric properties, the dye being preferably a reaction product of two spirit-soluble dyes which unite to produce a spirit-insoluble product. Thus sublimed flowers of sulphur are pasted with methanol and Spirit-Soluble Nigrosine SSB (*C.I.* 864) added and then with enough condensate of the dye—

with an organic base, e.g. triamylamine to completely precipitate the Nigrosine. The product after filtering and washing with methanol or warm water is a finely divided black powder suitable as an electrostatic dusting powder. C.O.C.

Diazoamino Compounds giving Azoic Dyes on Neutral Steaming (IV p. 109)
Colouring Polyacrylonitriles with Diarylmethane Dyes (VIII p. 115)
Rendering Wax-like Surfaces Receptive to Printing Ink, etc. (XIII p. 119)

X— SIZING AND FINISHING

Sizing of Spun Rayon Warps with special reference to Water-soluble Sizes
E. P. Sharman and T. A. Pask

J. Textile Inst., **47**, P 942–P 953 (Nov. 1956)

Small-scale work on miniature warps is described and data from commercial weaving trials are analysed. Disadvantages of starch sizes are enumerated, along with the advantages of the alternatives— natural vegetable products, modified starches and starch derivatives, cellulose ethers, and synthetic polymers— which are soluble in water. P.T.S.

Sizing of Yarns of Synthetic Fibres
L. Meier

Textil Praxis, **11**, 1207–1209 (Dec. 1956)

Sizing of polyamide, polyacrylonitrile, and polyester fibres with special reference to the use of Molvenin (Chemische Fabrik Grünau AG., Illertissen). B.K.

Finishing of Textile Fabrics
C. S. Whewell

J. Textile Inst., **47**, P 851–P 873 (Oct. 1956)

This lecture is primarily concerned with wool finishing, although the main theme, viz. the need for research into processes which are so well established as to be taken for granted, holds with equal force in almost all branches of finishing. The application of research to scouring, setting, crabbing, milling, felting, raising, blowing, pressing, and the production of dimensionally stable fabrics is discussed. Systematic data are urgently needed on the effect of altering the finishing routine on the appearance, handle, and usefulness of wool-type fabrics. The most convenient and effective ways of carrying out each finishing operation, of organising the flow of material from one part of the plant to another, and of manipulating the fabric on each type of machine must be worked out. This can be found only by scientific examination of existing procedures and detailed study of the fundamental operations involved. P.T.S.

Wash-and-wear Finishes on Cotton Fabrics
H. C. Borghetty

Amer. Dyestuff Rep., **45**, P 733–P 735 (24 Sept. 1956)

Application of crease-resistant finish is accompanied by a loss in strength. Melamine resins cause slight yellowing in the presence of chlorine. "Reactant-type" polymers act inside fibres and improve crease resistance without adversely affecting handle. Low-twist, loose-weave, and broken-pattern fabrics show least wrinkling. Soft thermoplastic acrylic resin improves crease-recovery without affecting strength, but some thermosetting plastic seems to be necessary for completely satisfactory crease-resistance. Careful control of conditions and catalysts is necessary for the best finish with the least loss of strength.

Silicone emulsions and non-ionic surface-active agents facilitate the actual finishing process and subsequent sewing. P.T.S.

Effect of Treatment in Aqueous Phenol Solutions on the Physical Properties of Secondary Cellulose Acetate Filaments
R. Jeffries and H. J. Wellard
J. Textile Inst., **47**, T 549–T 566 (Nov. 1956)

This paper continues and confirms earlier work (R. J. Marsden and A. R. Urquhart, *ibid.*, **42**, T 15 (1951)) on loss of strength of cellulose acetate when treated with delustring agents. It describes the effect of aqueous phenol and other swelling agents on strength of yarn, molecular structure of filaments, surface behaviour during extension, and the submicroscopic structure of the filaments (photomicrographs with crossed Polaroids, electron micrographs of surface structure, and X-ray photographs). Loss of strength is not directly related to degree of delustre, but is due to decrease in orientation. Lower breaking extension is caused by increased crystallinity. Best delustre with least loss of strength is obtained in the presence of cationic surface-active agent. Loss of strength is due more to internal change than to the surface cavities which are responsible for loss of lustre. P.T.S.

Finishing of Man-made Fibres
E. Brunnschweiler
Textil-Rund., **11**, 709–723 (Dec. 1956)

A condensed survey of dyeing methods, finishing, and auxiliaries. S.R.C.

Permanent Setting of Wool Fabric
S. Okajima and T. Inoue

III—Effect of the Temperature upon Crabbing
J. Soc. Textile Cellulose Ind. Japan, **12**, 732–736 (Oct. 1956)

The effect of temperature upon crabbing has been examined on a model plant in which the temperature was controlled at 77, 92, and 100°C. and the pH of the water was kept constant at 8·5, 9·2, and 9·5 in each case. The effect of crabbing was assessed by determining the decrease of the lustre of the fabric treated with steam. The results are—(1) The higher the crabbing temperature, the more lustrous is the fabric, and (2) the more effective is the permanent set at each pH value. (3) The effect of crabbing is highest at pH 9·2–9·5 even at the lower temperature. (4) The fabric is yellowed slightly by the treatment at 100°C. and at the higher pH, but crabbing at 77°C. does not show any yellowing even at pH 9·2–9·5.

IV—Permanency of the Lustre of Fabric pressed in the Air-dried State
Ibid., 736–739

Pressing the fabric in the air-dried state also raises its lustre, but the lustre is only temporary, and it falls almost completely to the original value when the fabric is released in the steam. This effect of pressing is a function of the moisture content in the fabric treated, and there is a critical moisture content (14–15%) in this experiment above which the lustre remains remarkably permanent. The permanency of the lustre is also high even when the fabric is slightly alkaline. AUTHORS

Silicones in the Textile Industry
A. A. Cook
Amer. Dyestuff Rep., **45**, P 771–P 775 (8 Oct. 1956)

Silicones are applied to cloth in the form of an emulsion and are made fast to washing and dry cleaning by a catalyst in a process analogous to the hardening of paint. Conditions are given for satisfactory application of silicones to fabrics. Silicones give high water-repellency, resistance to stains and spots, and improvement of wear resistance, tear strength, sewing qualities, and, with thermosetting resins, of crush resistance. Wide variations of handle can be obtained with different silicones combined with resins and softeners. Methods of estimating water-repellency and fastness of the finish to washing and dry cleaning are described, and the results of these tests given. Results of determinations of wear resistance and tear resistance of silicone-treated fabrics are cited. The application of silicones to wool, nylon–cotton, cotton, cellulose acetate, viscose rayon, and cellulose acetate–viscose rayon is described. P.T.S.

PATENTS

Sized Yarn from Green, Mechanically Broken Bark Fibre Material
Novivlas *BP* 763,716

Sizing Nylon, etc., with Hydrolysed Polyalkyl Acrylates
Monsanto *USP* 2,727,835

Cellulose Fibres containing Ether Groups and β-Propiolactone Substituents
U.S. Secretary of Agriculture *USP* 2,724,633

Cellulose fibres are etherified with, e.g. 2-amino ethyl sulphuric acid, to give 0·1–2·5 ether radicals per anhydroglucose unit, and are then treated with β-propiolactone at from 0°C. to slightly below their decomposition temperature until they contain 0·5–26·0% of combined β-propiolactone reaction products. The resulting fibres have increased diameter, decreased affinity for water and polar compounds and undiminished tensile strength. They have marked increase in resistance to dyes, heat, and acid degradation, and in ability to recover from creasing. C.O.C.

Unsaturated Cellulose Ether Fibres
U.S. Secretary of Agriculture *USP* 2,727,805

An alkali metal salt of a propargyl or allyl hydrogen sulphuric acid is dissolved in mercerising lye to yield a solution containing 5–15% of propargyl or allyl hydrogen sulphuric acid anions. The fibres are impregnated with this solution, dried and cured at 80–120°C. for 90–30 min. When a high degree of substitution is desired impregnation and curing are repeated several times. Alternatively, a relatively high degree of substitution can be obtained from a simple impregnation by wetting the cured impregnated material with water and recuring the wet material. C.O.C.

Scorching Characteristics of Fabrics (VI p. 112)
Effect of Humidity on the Tensile Behaviour of Grey, Scoured, Mercerised, and Chemically Modified Cotton (VI p. 113)
Effect of Resin Finishing on the Light Fastness of Vat Dyes (VIII p. 115)
Properties and Handling of Agilon Yarn (VIII p. 115)
Verel Acrylic Fibre (VIII p. 115)
Non-woven Sheet Material (XIII p. 119)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Optical Properties of Cellophane
E. F. Fahy and M. A. MacConaill
Nature, **178**, 1072–1073 (10 Nov. 1956)

Commercially produced Cellophane behaves as a half-wave plate; since the property is independent of the colour of the light used, it is concluded that it is a surface rather than a thickness effect. There is, nevertheless, a thickness effect in that Cellophane shows a definite coloration when it is viewed between polaroid sheets; this becomes more marked as the thickness increases. Polythene sheets exhibit the same properties, but to a lesser degree. No explanation of the half-wave plate effect is offered. J.W.D.

Periodate Oxidation of Water-soluble Methylcellulose and its Constituent Methylglucoses
G. C. Gibbons
J. Textile Inst., **47**, T 511–T 529 (Oct. 1956)

Sodium metaperiodate reacts with methylcellulose in three ways—(i) oxidation of glycol groups; (ii) chain scission, probably at the 4′-β-glucosidic link, causing a decrease in solution viscosity; and (iii) further reactions leading to a high degree of oxidation. Thus, use of periodate to estimate glycol content is unreliable, although (ii) is inhibited by acetate buffers and decreased in proportion to (i) by increase in concentration of the reacting solution, and (iii) is inhibited by acetic, butyric, and phthalic acids and decreased in proportion to (i) by raising the ratio of methylcellulose to periodate in the reaction mixture. Glycol group content measured in one experiment by periodate estimation was 0·21, against 0·187 by analytical estimation. Parallel experiments with constituent methylglucoses are described. P.T.S.

PATENTS

Coated Paper having Improved Bonding Strength
Hercules Powder Co. *USP* 2,727,837

Paper coated with a pigment dispersed in an aqueous solution of a gellable, water-soluble cellulose derivative and a setting agent for it and then dried has a printing surface consisting essentially of a film of pigment particles interspersed by the binder in which they are held on the paper. This removes the need for dipping and squeezing hitherto necessary in producing papers of this type. The coating composition has not a high alkali content and so the printed paper has a more uniform surface pH. C.O.C.

Metal-coated Cellulose Acetate-butyrate
Kodak *BP* 764,700

The surface of the cellulose acetate-butyrate article is coated with a thermosetting synthetic resin and then a metallic layer applied by vacuum coating. The metallic coating is then coated with a transparent epoxide-urea-formaldehyde resin and this in turn with a silicone resin. C.O.C.

Wood Finishes and Wood Finishing (V p. 112)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Amino Acid Composition of Different Proteins prepared from Skins and Hides of Indian Animals
S. M. Bose and B. M. Das
J. Amer. Leather Chem. Assocn., **51**, 647–656 (Dec. 1956)

Eight proteins, both fibrous and non-fibrous (collagen, reticulin, elastin, keratin, myosin, albumin, globulin, and mucoids) have been individually prepared in pure form from the skins and hides of Indian animals; each was characterised by means of total N and S determinations and by colour reactions. The component amino acids of each protein were quant. analysed by a two-dimensional paper-chromatographic technique. The results are reported, compared, and discussed. J.W.D.

Rôle of Crystallisation in Polymers and Proteins
P. J. Flory
Science, **124**, 53 (1956):
J. Amer. Leather Chem. Assocn., **51**, 619 (Nov. 1956)

A lecture reviewing the structure of fibrous proteins, their elastic properties, and their thermoelastic behaviour with special reference to collagen. Excellent 7-page review with 37 references. C.J.W.H.

Zirconium Tannage. VII—Direct Utilisation of Insoluble Zirconium Derivatives
I. C. Somerville and W. J. Rau
J. Amer. Leather Chem. Assocn., **51**, 542–555 (Oct. 1956)

Tannage may be effected by treating pelt with various insol. Zr cpd., of which sodium zirconium silicate is the most promising, in the presence of H_2SO_4 and neutral salts. The main advantage of the process is low cost. J.W.D.

New Developments in the Finishing of Leather
H. Loewe
Leder, **7**, 154–157 (1956):
J. Amer. Leather Chem. Assocn., **51**, 619–620 (Nov. 1956)

Buffed leather, for a natural surface, should have a multiple-layer finish. The first layer must be soft and elastic, and the following layers hard. A new cationic oil emulsion (made by BASF) is fixed by anionic vegetable or syntan leather and forms a suitable first or filling coat. A soft polymer dispersion will give the same effect. The covering coats may use one of three types of binders. The most important protein type is casein, which is fast to acetone and hot pressing but must be combined with a plasticiser to avoid a hard, brittle film. The plasticisers may be lost during storage of the leather and have other disadvantages. Good buffed leather could not be made without polymerised dispersions. These need no plasticiser and can be used in high concentrations without being too viscous. One of the oldest products of this type is the polyacrylic ester Corialgrund O conc. The properties of such an ester depend on the alcohol constituent. The higher the mol. wt. of the alcohol, the softer the polymer film. Also the lower the D.P., the softer and stickier the film; while the higher the D.P., the firmer and more rubber-like the film is. A series of vinyl compounds are also used as binders. To be used in finishing, binding products must meet certain requirements both in the shoe factory and during use of the leather article, such as satisfactory hot pressing and compatibility with pigment systems. Some information on the suitability of a product can be obtained by examining a film made by allowing the water to evaporate from a layer of the dispersion. A new protein-type binder, Luron, combines the good properties of casein and plastic binders. It is a colloidal solution of a protein-like substance that forms an elastic film which is not thermoplastic and is insoluble in acetone. This product is hardened with HCHO, but so slowly that the HCHO is mixed with it. The first coat should contain no HCHO in order to avoid a bad effect on the leather grain. Glyoxal may be used instead of HCHO if one wishes to avoid the odour of the latter during drying of the leather. C.J.W.H.

Pigments and Finishing Agents
P. Penaud
Bull. Assocn. Française Chim. Ind. Cuir, **18**, 203–210 (Sept. 1956)

A brief discussion of the pigments and binding media available, and of the principles underlying their selection in the formulation of leather finishes for various purposes. The main emphasis is upon casein as binder; synthetic resins are considered only as emulsions, and the possibility of their use in combined finishes with casein is briefly treated. J.W.D.

Application of Colorants to Leather
I—Chemical and Physical Nature of Leather Fibres in relation to Dyeing
R. Stubbings
J. Amer. Leather Chem. Assocn., **51**, 514–519 (Oct. 1956)

II—Colouring of Leathers on which Penetration is desired
J. A. Casnocha
Ibid., 520–523

III—Spray or Brush Dyeing of Leather
C. H. Geister
Ibid., 523–526

IV—Dyes and Dyeing Processes for Special Properties of Colour
J. S. Kirk
Ibid., 526–529

Batik Art in Leather Finishing in Indonesia
S. Partroesti and E. Gergely
Leder, **7**, 134–136 (1956):
J. Amer. Leather Chem. Assocn., **51**, 625–626 (Nov. 1956)

"Batik leather" has recently aroused interest not only in Indonesia but in other countries where national costumes are worn. It has appeared in Europe and in the U.S.A., where lately a kind of screen printing was used to produce patterned upper leather from pigskins. In Indonesia the old "Foldik" leather method, which produces colour patterns, has lately been promoted for use in cottage tanneries by "modernised" procedures. A goatskin, tightly pleated and fastened to a board with copper nails at each end, is coloured with different dyes, which are applied to the edges of the folds only, to produce a pattern design. The skins are then unfolded, stretched, dried, dressed with a slightly coloured top dressing, and boarded by hand. A description is given of the batik process for dyeing fabrics, in which the cloth is waxed by hand to form a design and dyed (in the unwaxed areas), and the wax is removed by boiling the cloth in water. For leather, the wax is applied either by a brass pattern or through a stencil. C.J.W.H.

Binding Capacity for Hydrochloric Acid of Hide-powder in Sodium Chloride Solutions
P. M. Heertjes and O. H. Djoen
J. Soc. Leather Trades Chem., **40**, 346–360 (Nov. 1956)

A method based on the Donnan membrane theory is given for calculating the acid-binding properties of collagen as a function of pH. The hide-powder was agitated with a soln. of HCl and NaCl at 20°c. until equilibrium was reached; the swollen hide-powder was then filtered off, and the following were determined in the filtrate—[H], [Cl], pH, and peptised collagen (by Kjeldahl N determination). The weight ratio of solid to liquid, and the concn. of NaCl and HCl, were treated as variables. The amount of water

retained by the centrifuged hide-powder was also determined. When [NaCl] < 1%, the ratio between the activities of dissolved Na, Cl, and H = 1. Three distinct water phases may exist—(1) the outer phase, (2) that in the gel, containing no dissolved substances, and (3) the inner water phase (in the gel) in which the concn. differ from those in phase 1. When the above ratio = 1, the differences between phases 1 and 3 disappear. The proton-binding capacity (z) of collagen is related to [H] by the expression—

$$z = H_{total} - \frac{[Na]_{total}}{[Cl]_u - [H]_u} \cdot [H]_u$$

where the subscript "u" refers to concn. in the outer phase. The real, corrected binding capacity (z'') is given by—

$$z'' = z + [Na]_b \cdot \frac{[H]_u}{[Cl]_u - [H_u]}$$

where "b" refers to the outer phase. Though z increases with increasing activities, a max. constant value was not found, probably because of the weak proton-binding capacity of the "imino" groups. Protons are bound to the basic groups of collagen in accordance with the equation—

$$-NH_2 + H^+ \rightleftharpoons NH_3^+$$

or, if salt linkages occur in the collagen, according to—

$$-NH_3^+ \}\text{-OOC}- + H^+ \rightleftharpoons -NH_3^+ + COOH$$

J.W.D.

Improvement of the Fastness of Dyed Pelts
C. Rouanet
Bull. Assocn. Française Chim. Ind. Cuir, **18**, 195–202 (Sept. 1956)

Dye fastness depends upon the nature of the tannage used, the type and the structure of the dye(s), and the method of application. These factors are briefly discussed from the viewpoint of improving the fastness of pelt dyeings to light, to wet treatments (action of water, perspiration, washing, and degreasing agents), and to wet and dry rubbing. J.W.D.

Absolute Configuration and Optical Rotation of Folded (α) Polypeptides
A. Elliott and B. R. Malcolm
Nature, **178**, 912 (27 Oct. 1956)

Measurements with a reflecting optical diffraction spectrometer suggest that α-helices in poly-L-alanine and presumably other L-amino acid polymers are right-handed. W.R.M.

Optical Rotation of the α-Helix in Synthetic Polypeptides
A. Elliott, W. E. Hanby, and B. R. Malcolm
Nature, **178**, 1170 (24 Nov. 1956)

Measurements of the optical rotation of DL-leucine polypeptides in benzene suggest that L-polypeptides form right-handed helices. It is possible that the optical rotation of an α-polypeptide can be treated as the sum of contributions from the helix core and the asymmetric centres. W.R.M.

Practical Considerations in Waterproofing Leather (XIV p. 120)

XIII—RUBBER; RESINS; PLASTICS

"Living" Polymers
M. Szwarc
Nature, **178**, 1168–1169 (24 Nov. 1956)

In a polymerisation process which does not involve a termination step the polymeric molecules may be regarded as "live". If monomer is exhausted, growth is interrupted, but the polymer may grow if additional monomer is supplied. Treatment with reagents leading to a termination step will "kill" the polymer. Experiments illustrating these concepts are described, and a novel method is proposed for the preparation of block copolymers. W.R.M.

Reaction Mechanisms in the Photochemical Redox Polymerisation of Acrylonitrile
S. Neue
Faserforsch. und Textiltech., **7**, 485–489 (Nov. 1956)

The photochemical polymerisation of acrylonitrile in aqueous solution is studied, using the redox system oxalic acid–ferrous salt–hydrogen peroxide as initiator. The velocity of formation of nuclei (approximately the reciprocal of the period of induction) is proportional to a power, between 0·5 and 1, of $[Fe^{2+}]$. The velocity of polymerisation is proportional to the concentrations of ferrous ion and acrylonitrile. The mol. wt. of the polymer is inversely proportional to $[Fe^{2+}]^{\frac{1}{2}}$ and directly proportional to the concn. of monomer. W.R.M.

Polycondensation of Benzyl Chloride catalysed by Stannic Chloride
L. Valentine and R. W. Winter
J.C.S., 4768–4779 (Dec. 1956)

The kinetics of the polycondensation of benzyl chloride (I) to form polybenzyl, catalysed by stannic chloride, show that for each mol. consumed, one mol. of HCl is evolved, and that up to a catalyst concn. of 0·6M. the rate is directly proportional to the catalyst concn.; it then attains a max. at ca. 1·1M., and at higher concn. becomes inversely proportional to the catalyst concn. The reaction is of the first order with respect to I, the activation energy is 7·9 kcal./mole, and the D.P. increases rapidly with extent of reaction at first, but becomes const. at extents of reaction $\geqslant 30\%$. At high conversions, the D.P. is independent of temp. or catalyst concn. The reaction appears to be a polycondensation process proceeding by a chain reaction; probable reactive intermediates are discussed. Infrared and ultraviolet spectra of the polymers show identity of structure consistent with the highly subst. polybenzyl structure proposed by Haas, Livingston, and Saunders (*J. Polymer Sci.*, **15**, 503 (1955)), i.e. with a nucleus of almost completely subst. benzene rings and a periphery of pendant benzyl groups. H.H.H.

PATENTS

Multi-coloured Patterned Sponge Rubber
"Semperit" Österreichisch-Amerikanische Gummiwerke
BP 765,399

A mixture of pieces of differently coloured raw rubber compositions containing substances which release a gas on heating, each piece having been treated with an adhesion inhibiting agent, e.g. talc, is heated in an open container to first cause the pieces to fuse together, then to cause the gas to be evolved and finally to vulcanise the mass. C.O.C.

Non-woven Sheet Material
DuP *BP* 763,605

Material composed of, by wt., 20–85% of non-woven matted fibres and 80–15% of an extensible polymeric binder, the fibres and binder being chemically dissimilar and incompatible, and having a surface layer where the fibres are all < 0·2 denier/filament and a base layer where the fibres are all > 1·0 denier/filament, have very satisfactory tear and tensile strength, flexibility and uniform extensibility and in addition have very good resistance to scuffing. C.O.C.

Artificial Leather
DuP *BP* 763,604

A water-vapour impermeable, non-extensible material consisting of particles or fibre is impregnated with a water-impermeable, extensible thermoplastic polymer and then hot pressed to form a sheet. The polymer should make up 30–70% by weight of the final product. The sheet is then stretched and finally relaxed to yield an artificial leather which is permeable to water vapour. C.O.C.

Rendering Wax-like Surfaces Receptive to Printing Ink, etc.
C. G. Lemon *BP* 765,545

Polyethylene or similar wax-like material has its surface exposed to a high voltage glow or corona discharge of electricity along a narrow line at a voltage and for a time sufficient to so modify the surface that printing ink, paint etc. will adhere to it. C.O.C.

Patent Leather Finish on Polyvinyl Chloride Sheeting
DuP *BP* 763,646

Polyvinyl chloride sheeting is bonded to a film of dissimilar material by passing the two through pressure rolls

so that there are no air spaces between the sheeting and the film. One of the rolls is hot or the sheeting is heated immediately before laminating. The adhesion between the sheeting and film must be such that the product can be rolled on a 3 in. diameter core without the bond breaking but must be low enough to permit the film to be stripped from the sheet without marring the surface finish. After the film is removed the sheeting has a smooth mirror-like finish. C.O.C.

Imparting an Antistatic Finish to Polystyrene
Westinghouse Electric Corpn. *USP* 2,727,831

The article is treated with conc. H_2SO_4, drained, rinsed and neutralised. The treated article no longer accumulates an electrostatic charge and no longer attracts dust and grit even when exposed to dusty atmospheres for prolonged periods. The finish is resistant to washing and cleaning. C.O.C.

Microscopy of Heterogeneous Polymeric Systems (V p. 120)

Use of Irradiated Polymers as Initiators of Polymerisations (VI p. 113)

XIV— ANALYSIS; TESTING; APPARATUS

Determination of the Character of the Charge on Auxiliary Products
G. Otto

Leder, **7**, 145–148 (1956): *J. Amer. Leather Chem. Assocn.*, **51**, 618 (Nov. 1956)

It is important to know the nature of the surface charge of auxiliary tannery products because this influences their behaviour. Products with opposite charges will precipitate (or form a cloudy solution) when mixed. Nekal BX (BASF) can be used to test for cationic materials, and Levogen WW (FBy) for anionic products, but the test solution must be added drop by drop, because an excess of either the reagent or the material being tested will cause the precipitate to redissolve, and the cloudiness may not be observed if the addition is too rapid. The Epton titration method (*Trans. Faraday Soc.*, **44**, 226 (1948)) is described in which Methylene Blue and chloroform are used as indicators. With an excess of an anionic product the chloroform layer is blue, but with an excess of cationic product the aqueous layer is blue. The BASF can now supply indicators to test either for cationic substances (Indicator A, which is identical with Corialgrund O conc.) or for anionic substances (Indicator K). A few ml. of the unknown solution is added to each of two test-tubes, and a few drops of the indicators are added. Cloudiness with A indicates that the solution contains a cationic substance, while cloudiness with indicator K indicates an anionic substance. If neither tube is cloudy, the substance is non-ionic. With these indicators one can rapidly determine the charge on wetting, tanning, dyeing, or oiling agents. The method fails only if salts of multivalent metals are present in more than trace amounts. In the discussion of the paper it was pointed out that the test may fail if protective colloids are present and also with some special products such as mixtures of non-ionic substances with either anionic or cationic substances. C.J.W.H.

Absorption Spectra of Leuco-base Dyes in Acid Solutions and in the Solid State. II— Leuco Bases of Thiazine and Oxazine Dyes in the Solid State
A. T. Vartanyan

Zhur. fiz. khim., **29**, 1447–1445 (1955): *Chem. Abs.*, **50**, 16383 (25 Nov. 1956)

A description of a method of preparing solid films of the leuco bases of thiazine and oxazine dyes is followed by the absorption spectra of the leuco bases of Lauth's Violet, Methylene Blue, Thionine Blue, and Capri Blue. The spectra show that in the initial stages of regeneration, the absorption spectra of solid solutions of the dyes in the leuco bases are similar to the spectra of the liquid solutions. The concentration of the regenerated dye is greater in the solid solutions; dimeric forms of the dye are formed. The rôle of the H_2O molecule and the changes in the spectra in the transition from solution to the solid state are discussed. C.O.C.

Absorption Spectra of Sublimed Dye Layers
A. T. Vartanyan

Zhur. fiz. khim., **30**, 1028–1043 (1956): *Chem. Abs.*, **50**, 16393 (25 Nov. 1956)

Study of absorption spectra of solid dyes in layers formed by sublimation offers the advantages over layers obtained by solution evaporation of being in perfect shape for spectrophotometric measurements, of being applicable with dyes insoluble in readily vaporised solvents, and of giving the dyes an additional purification by sublimation. The disadvantages are that not all dyes can be sublimed without decomposition and that some dyes form non-dispersing deposits. Measurement of the absorption spectra of 48 dyes and comparison of the absorptions of the two types of layers enabled the dyes to be divided into three classes— (*a*) dyes whose absorption spectra are the same with both types of layer and with absorption curves differing from those of their alcoholic solutions; (*b*) dyes whose absorption spectra differ according to the way the layer is formed and whose sublimed-layer absorption spectra are similar to those of their alcoholic solutions; (*c*) dyes giving different absorption spectra depending on the temperature of sublimation. C.O.C.

Circular Paper Chromatography of Some Food Dyes
A. Venturini and L. Zanoni

Boll. Lab. chim. provinciali (Bologna), **7**, 16–19 (1956): *Chem. Abs.*, **50**, 17223 (25 Nov. 1956)

Two glass dishes 30 cm. in diameter and 7 cm. deep are used. The upper dish has in its centre a 5-cm. opening through which the tip of a pipette or a syringe containing the solvent is introduced. The amount of solvent released at each test on the filter paper was 2·5 ml. The paper was subsequently oven-dried for 8 min. at 100°c., then developed for 2 hr., and again oven-dried. The dyes used were C.I. Food Yellow 1 and 4, C.I. Food Orange 4, C.I. Food Red 14 and 15, C.I. Basic Orange 2, Victoria Red, Sudan II, and Eosin Yellow. The best solvents for them were ethyl alcohol at 30°c. and water-saturated butanol. The R_f values obtained by this method are in good agreement with those obtained by other methods. C.O.C.

Spectrophotometry of Granulated Materials, with particular reference to Blood Corpuscles
G. F. Lothian and P. C. Lewis

Nature, **178**, 1342–1343 (15 Dec. 1956)

Synthetic Linear Polymers. II— Viscometric Determination of the Molecular Weight of Polyamides in Aqueous Chloral Hydrate
I. Rusznák, I. Géczy, and A. Ady

Faserforsch. und Textiltech., **7**, 490–493 (Nov. 1956)

The relationship between intrinsic viscosity [η] and D.P. for 6-nylon in 70% aq. chloral hydrate is—

$$[\eta] = 0{\cdot}01996\ (\text{D.P.})^{0{\cdot}78}$$

W.R.M.

Method for Quick Ascertainment of Penetration of Aniline Dyes into Leather
P. Kozina

Koza i Obuca [*Leather and Footwear*], **4**, 237–242 (1955): *J. Amer. Leather Chem. Assocn.*, **51**, 624 (Nov. 1956)

The test is made by using chromatographic paper, employing dilute NH_4OH as solvent. C.J.W.H.

Practical Considerations in Waterproofing Leather
C. W. Mann

J. Amer. Leather Chem. Assocn., **51**, 634–646 (Dec. 1956)

A new machine is described which applies a tapping and rubbing action to a leather specimen in contact with a wet surface; this causes faster penetration (especially with the more resistant leathers) than the Maeser test (*ibid.*, **42**, 390 (1947)). There is good agreement with the results of glove- and shoe-wearing trials. The water-resistant characteristics of leather for different applications are discussed. There is a sketch of the apparatus. J.W.D.

Simple Method for the Investigation of Peptides containing Cystine and/or Cysteine
N. R. Ling

Nature, **178**, 1054–1055 (10 Nov. 1956)

The method, which is fully described, is based on the fact that cysteine and cystine are held on a cation-exchange resin, but cysteic acid is not. J.W.D.

Microscopy of Heterogeneous Polymeric Systems (V p. 111)

Silicones in the Textile Industry (X p. 117)

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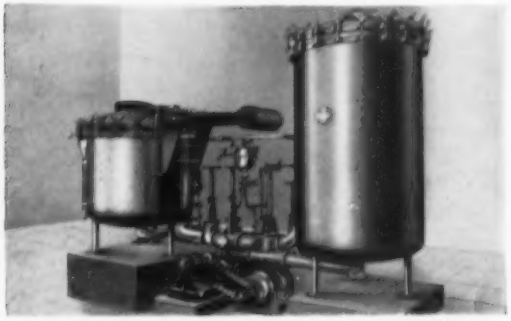

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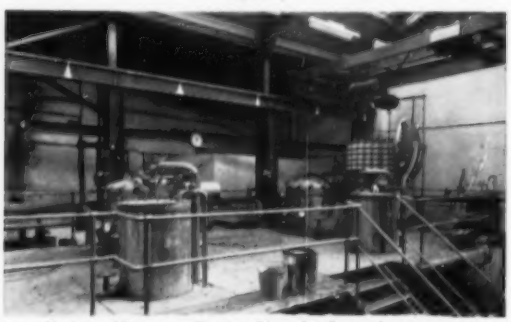

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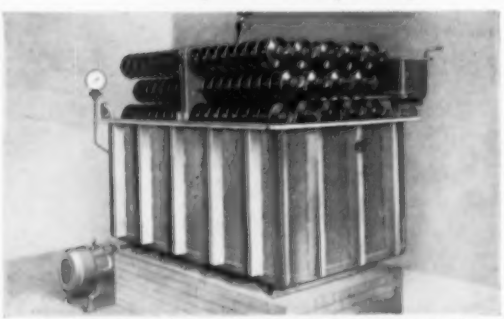

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MEMBERS' CHANGES OF ADDRESS— *continued from page viii*

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Glover, N., formerly of Wortley Low Mills, Leeds, Yorkshire, to c/o J. K. Howarth, 3 Greenville Avenue, Leeds 12, Yorkshire

Grayson, Edward H., formerly of 2511 Lucena Street, Charlotte 6, North Carolina, U.S.A., to Carbic Color & Chemical Co. Inc., 123 East Griffith St., Charlotte 3 N.C., U.S.A.

Hadfield, H. R., formerly of 5 Banks Lane, Stockport, Cheshire, to 4 Corbor Road, Stockport, Cheshire

Hallows, L. B., formerly c/o W. R. Grace & Co. Ltd., Casilla 2488, Fabrica Inca, Lima, Peru, S. America, to 85 Easton Avenue, Apt 4D, New Brunswick, N.J., U.S.A.

Ingamells, W. C., formerly of 952 Kingsway Extension, East Didsbury, Manchester, to 33 Saxton Avenue, Bessecar, Doncaster, Yorkshire

Isenring, J. L., formerly of Dionne Spinning Mills Co., Box 464, St George, Beauce, Quebec, Canada, to Box 464, Ville St Georges Bce, P.Q., Canada

Kaye, H., formerly of 14 Barfield Drive, Yeadon, near Leeds, to The Bungalow, Britannia Road, Morley, near Leeds

Kuhl, C. W., formerly of 451 Washington Street, New York, N.Y., U.S.A., to Carbic Color & Chemical Co. Inc., 451 Washington Street, New York 13, N.Y., U.S.A.

Levi, S. S., formerly of 5 Fairhazel Mansions, Fairhazel Gardens, London N.W.6, to Federal Dept. of Commerce and Industries, Institute of Applied Technical Research, Lagos, Nigeria

Marsden, J., formerly of "Rowlands", 7 Cranford Avenue, Whitefield, Lancashire, to 115 Victoria Street, Magog, Quebec, Canada

Meston, Wm., formerly of Grandholme Works, Woodside, Aberdeenshire, Scotland, to Rosemount, Peterculter, Aberdeenshire, Scotland

Midwinter, W. S., formerly of 67 Fairlands Road, Limefield, Bury, Lancashire, to "Lismore", 22 Bury and Bolton Road, Radcliffe, near Manchester

Nicholls, C. H., formerly c/o C.S.I.R.O., Wool Textile Research Labs., Belmont Geelong, Aust., to School of Textile Technology, N.S.W. University of Technology, P.O. Box 1, Kensington, New South Wales, Australia

Noble, P. G., formerly of 9 Albany Place, Dumfries, Scotland, to R. P. Lawson & Co. Ltd., Rodney Street, Oldham Road, Manchester

Pensa, Ildo, formerly c/o Pensione Torquati, Via Giosue Carducci 2, Rome, Italy, to Via Bisagno 24, Rome, Italy

Pineger, R. N., formerly of Works Dyehouse, Clayton Aniline Co. Ltd., Clayton, Manchester, to 14 Ashbourne Road, Hazel Grove, Cheshire

Rao, D. K., formerly of 10th Main Road, 17th Cross Road, Malleswaram P.O. Bangalore, South India, to No. 18 Tenth Main Road, 16th Cross Road, Malleswaram P.O., Bangalore, India

Raynes, J. L., formerly of Amberside, Heage Road, Ripley, Derbyshire, to c/o Stevensons (Dyers) Ltd., Amber Dye Works, Ambergate, Derbyshire

Rhodes, P., formerly of 75 Wavertree Drive, Leicester, to P.O. Box 447, Huntingdon P.Q., Canada

Rosner, Stanley S., formerly of Nina Dye Works Co. Inc., York, Pa., U.S.A., to The United Piece Dye Works, York, Pa., U.S.A.

Seddon, H. P., formerly of 30 Whitham Street, Ashton-under-Lyne, to 84 Derby Road, Ashton-under-Lyne, Lancashire

Slater, J., formerly of Kary Hill, Downpatrick, Co. Down, Northern Ireland, to The Cottage, Ballygeegan, Killinchy, Co. Down, Northern Ireland

Sykes, J. A. W., formerly of 27 Yew Street, Fartown, Huddersfield, Yorkshire, to 39 Woodhouse Lane, Brighouse, Yorkshire

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Tomlinson, E., formerly of "Methwold", Dickens Lane, Boynton, Stockport, Cheshire, to "Towers Wall", South Park Drive, Poynton Park, Poynton, Stockport, Cheshire

Waterhouse, J. R., formerly of 2 Gynsill Court, Gynsill Lane, Anstey, near Leicestershire, to 34 Desford Road, Kirby Muxloe, Leicestershire

Whittaker, Dr. C. M., formerly of 1 Weaponness Park, Scarborough, Yorkshire, to Red Lea Hotel, Scarborough, Yorkshire

Williamson, A. J., formerly of 41 Allen Street, Allerton, Derby, to 12 Springfield Road, Chaddesden, Derby

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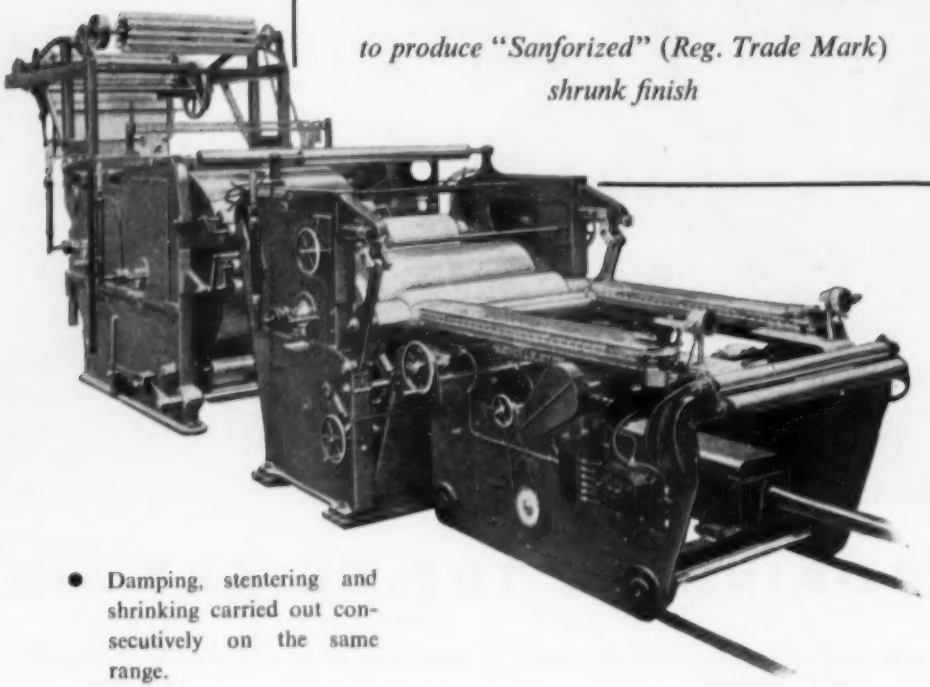

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